LA-UR-07-1371

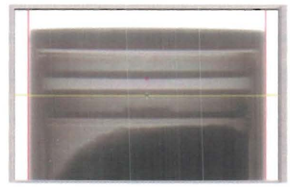
Surveillance & Monitoring Annual Meeting
January 23-25, 2007
766-H, Room 2138

Tuesday, AM		
7:00 – 8:00	Badge office for those going on tour (30 minute drive to 766-H)	
8:30 – 8:45	***Welcome/Introductions***	A. Gunter
	Shelf Life Studies	
8:45 – 9:05	Large Scale Surveillance Update 20 min	J. Berg
9:05 – 9:35	SRS Corrosion studies 30 min	J. Duffey
9:35 – 10:05	11589 Action Plan Overview 30 min	J. Berg
10:05 – 10:35	Oxygen generation: Correlations observed with waters of hydration 30 min	E. Garcia
10:35 – 11:05	Scope and status of SRS Destructive Evaluation Operations 30 min	K. Dunn
11:05 – 12:00	**Lunch**	
Tuesday, PM	**TOUR OF KIS, SRNL, KAMS – Tuesday afternoon**	
	Side Meetings – Tuesday afternoon	
	• Narlesky and SRS Surveillance Engineers to review prompt gamma software and analysis	
	• Corrosion studies technical exchange (Lillard, Zapp, Berg, Garcia, Livingston, Veirs?, Dunn)	
Wednesday	***Special Topics***	
8:30 – 9:30	Update from AWE 60 min	S. Shaw
	Interim Packaging, Storage, and Surveillance	
9:30 – 9:45	**Break**	
9:45 – 11:45	Presentation and Discussions on O2/H2 combustion 120 min	J. Shepherd
11:45 – 12:45	**Lunch**	
	Shelf Life Studies	
12:45 – 1:45	Small Scale Surveillance Gas Analysis – with tie to expected pressures and gas compositions in field containers 60 min	K. Veirs
1:45 – 2:15	Material deliquescence 30 min	K. Veirs
	Special Topics	
2:15 – 2:30	PAT-1 and PuFe eutectic issues 15 min	R. Mason
2:30 – 2:45	**Break**	
	Field Operations and Surveillance	
	Review of FY06 Surveillance data	
2:45 – 3:15	SRS 30 min	C. McClard
3:15 – 3:45	Hanford 30 min	T. Venetz
3:45 – 4:05	LLNL20 min	S. Mayhugh, K. Dodson
4:05 – 4:25	LANL20 min	R. Mason
4:25 – 4:55	2005-1 Status of Interim Storage and Packaging Manual 35 min	G.D. Roberson

Thursday	***Site Operations***	
8:30 – 9:30	LANL Packaging Program: LANL Program Update; Comprehensive Plan; 2000-1 update 60 min	P. Smith, L. Remerowski, W. Punjak
	Database and Analysis	
9:30 – 9:50	Rebinning of Items – Overview of LA-14310 20 min	L. Peppers
9:50 – 10:20	Discussion on 2008 sample selection and beyond 30 min	
10:20 – 10:30	**Break**	
10:30 – 11:15	SRS ISP Database Efforts: LANL / SRS Transition; Completed Ongoing Efforts; Future Plans 45 min	B. Cheadle, G. Friday, T. Paul
11:15 – 11:45	Prompt-gamma update 30 min	J. Narlesky
11:45 – 12:00	Pressure calculations in ISP Database 15 min	L. Peppers
12:00 – 1:00	**Lunch**	
1:00 – 2:00	Linkage of MIS / ISP Databases: 60 min	J. Narlesky / L. Peppers
	Verification of MIS items representation	
	Master-Table based on Representation Documents	
2:00 – 4:00	***MIS – WG Discussions***	
	ISP Steering Committee – Meeting wrap-up 120 min	A. Gunter, G.D. Roberson, MIS-WG
	AOP Discussions	
	Side Meetings – Thursday afternoon	
	SRS database (Cheadle) and LLNL Database engineers	

PFP Fiscal Year 2006 3013 Surveillance Activities, Including ISP Field Surveillance.

Presented to
The MIS Working Group
FY 06 Annual
MIS Meeting
Savannah River Site



T.J. Venetz
Fluor Hanford

S. E. Clarke
DOE/RL

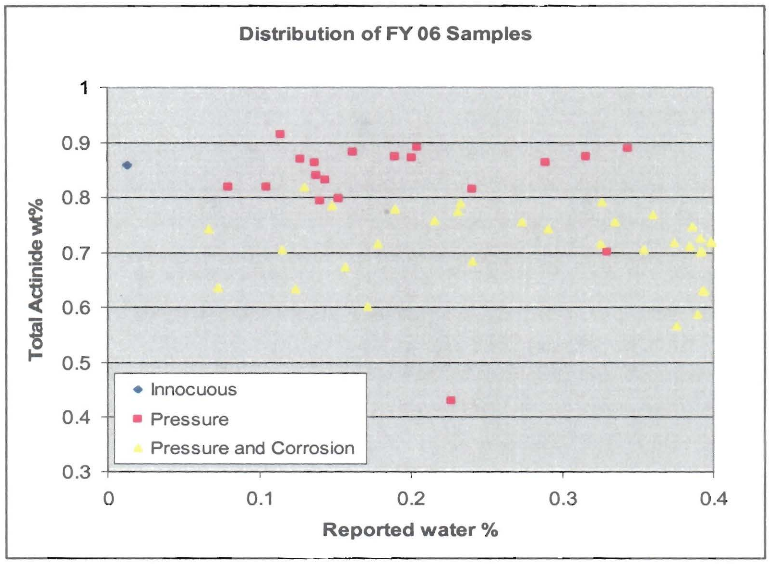
January 23-25, 2007

Fluor Hanford

Hanford FY 06 Samples

- **24 ISP samples selected by the Working Group**
  - 18 Randomly Selected
  - 6 By Engineering Judgment
    - Pure oxides, Rocky Flats Oxide (ARF), Oxide from impure solutions all with "high water"
- **29 additional PFP samples**
  - 13 items like MIS item 11589, with flammable gas issue
    - All those judged "most similar",
    - Those judged potentially similar with over 0.3 wt% water
  - 4 items with highest post sample weight gain
  - 6 ARF items with detectable weight gain or greater than 9 grams total water
  - Other Items processed in "wet" line with high reported water content
  - 4 repeats from FY 05 sample
- **Prompt Gamma Analysis on items judged "Potentially Similar" to MIS item 11589**

Sample Makeup

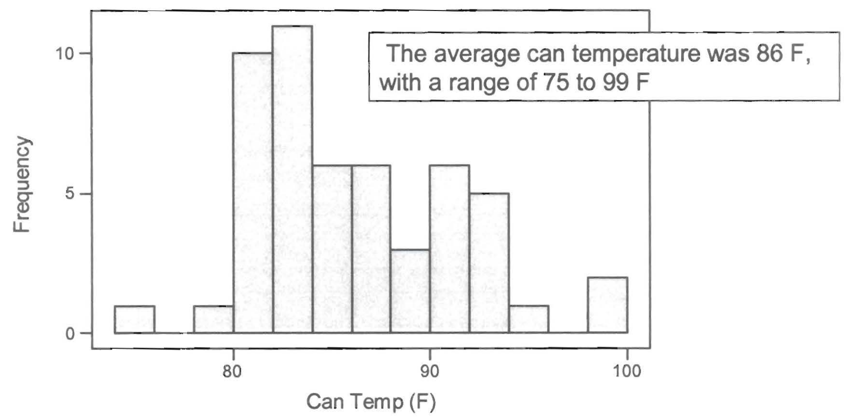


Results Obtained for

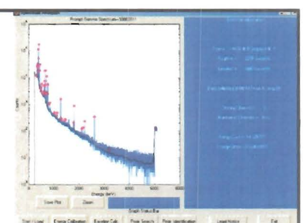
- Temperature (can and vault ambient)
- Contamination Status – All nondetectable
- Dose Rates
  - Individual maximums - 270 mr/hr gamma, 110 mr/hr neutron
- Visual Inspection – No abnormalities noted
- Gross Weight Change
  - All within 2 grams/none detectable
- Lid Deflection Change – follows
- Prompt Gamma Analysis - follows

Temperature Results

Histogram of Can Temperature (F)



Prompt Gamma Results



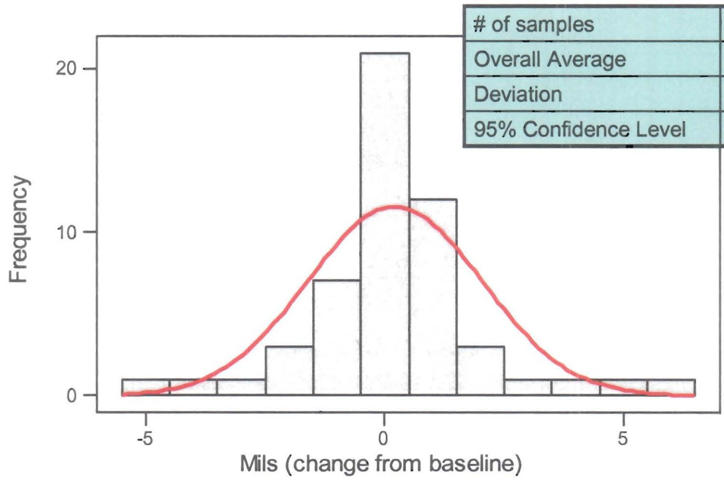
- **ISP and PFP Surveillance samples**
  - 28 items have Chloride, 1 has strong Fluoride
  - All were correctly assigned to Pressure and Corrosion Bin
  - One ARF item originally binned as Pressure and Corrosion shows only weak Fluoride
  - Pure Oxide and Mixed Oxide samples show either no impurities or only trace Impurities (Al, Mg, Na)
  - 2 ISP samples found to have anomalous PG spectra and have been rerun
- **Items Potentially Similar to 11589 (96 were identified for Hanford)**
  - Of 48 analyzed so far, only 5 are judged similar by Prompt Gamma
  - Prompt Gamma analysis still ongoing for the remaining 38 items

Radiography

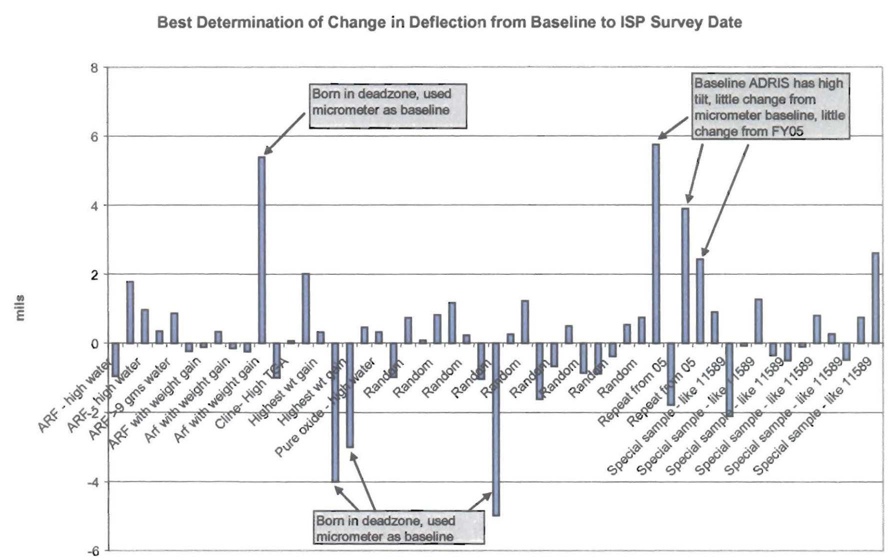
- **All containers inspected with new ADRIS ver 1.3 allowing multiple angle imaging to mitigate deadzone issue**
  - Traditional ADRIS – 4 angle average
  - Select View – pick best of existing 2 views - 4 angles
  - Best View – Pick best of 8 views – 16 angles
- **Requires careful evaluation off-line**
  - Writeup on best determination methodology for each can included in report on PFP Fiscal Year 2006 Surveillance Activities (HNF-31775) and in surveillance database

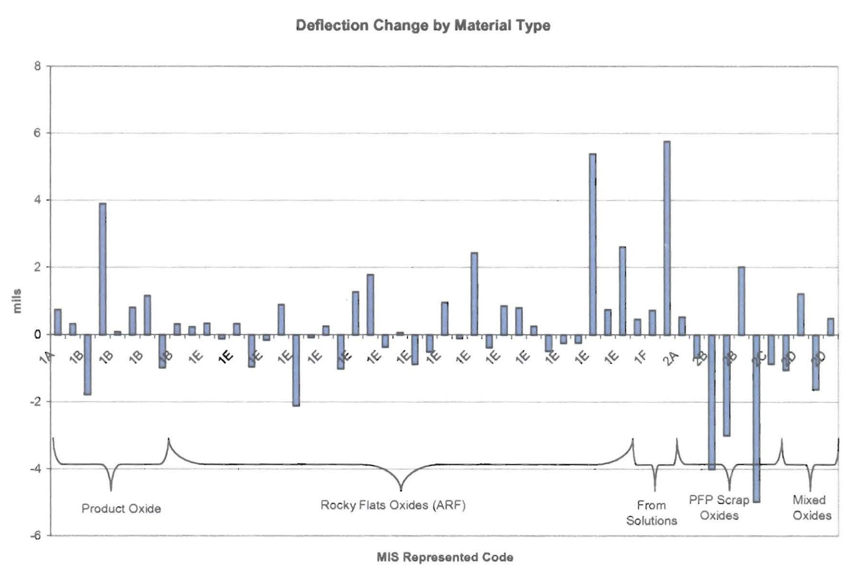
Radiography

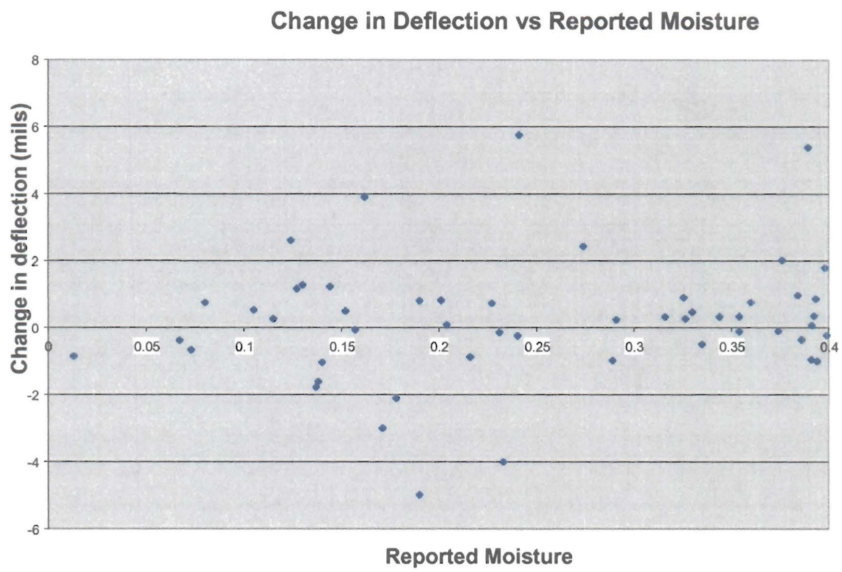
Histogram of Best Determination of Deflection Change -FY06



# of samples	53
Overall Average	+0.2
Deviation	1.8
95% Confidence Level	-0.3 to +0.7


Best Determination of Change in Deflection from Baseline to ISP Survey Date
mils
8
6
4
2
0
-4
-6
Born in deadzone, used micrometer as baseline
Baseline ADRIS has high tilt, little change from micrometer baseline, little change from FY05
Born in deadzone, used micrometer as baseline
ARF - high water
Arf with weight gain
Arf with weight gain
Highest wt gain
Random
Random
Random
Random
Repeat from 05
Special sample - like 11589

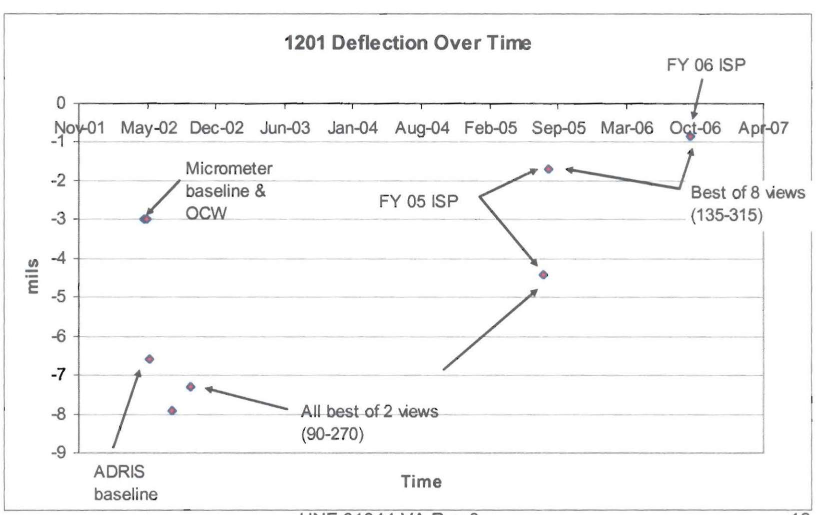

Deflection Change by Material Type
mils
8
6
4
2
0
-2
-4
-6
1A
1B
1E
1F
2A
Product Oxide
Rocky Flats Oxides (ARF)
From Solutions
PFP Scrap Oxides
Mixed Oxides
MIS Represented Code



HNF-31944-VA Rev 0 11

Container with highest overall change

- Fairly pure mixed oxide precipitated from mixed nitrate solution
- Water less than 0.25%, low net weight

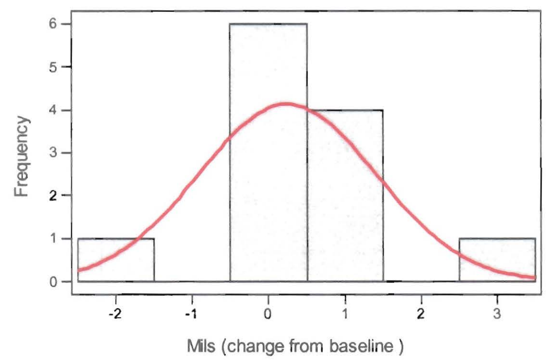


HNF-31944-VA Rev 0 12

Items Similar to MIS item II589 Show no Detectable Pressure

- None was expected based on sample water % and can loading
- Each loading parameter about ½ of the Shelf-life test can

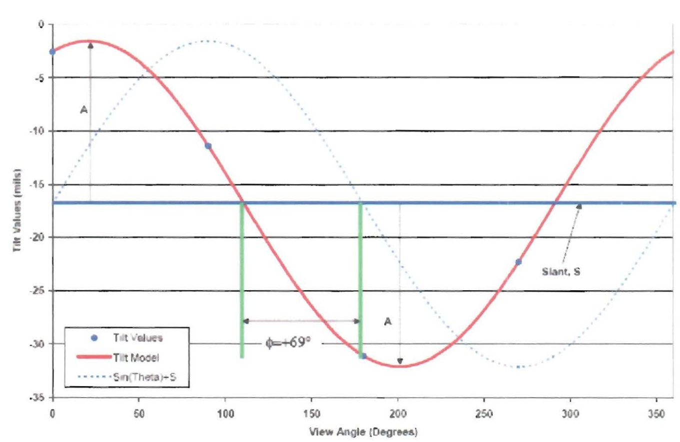
Histogram of deflection change, items similar to 11589



# of samples	13
Overall Average	+0.25
Deviation	1.2
95% Confidence Level	-0.5 to +1.0

Radiography – Optimal View Model

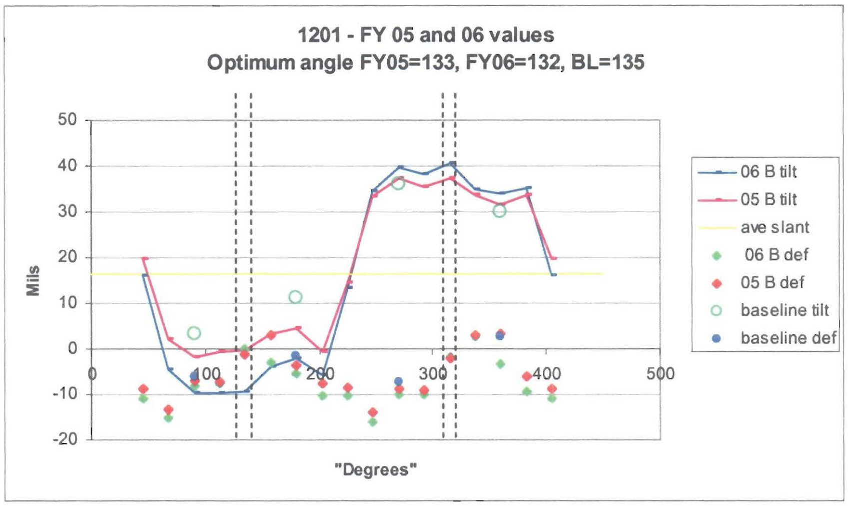
Slant, Amplitude and Phase Determination



Optimal View Model developed by SRS

Tilt values follow sinusoid pattern, with optimal angle found at maximum tilt

Tilt Values Follow Predicted Model



Radiography Conclusions

- Generally no sign of detectable pressurization, similar to last years results
- Cans that exceed detection level limit are explainable
- Some resurvey recommended for next year
- None of the items in subgroup similar to MIS item 11589 show detectable pressurization, consistent with expectations
- ADRIS 8 angle radiography mitigates deadzone issue

FY 07 Plans

- Continue with FY07 NDE, with selection of additional samples, repeat some FY06 samples
- Continue/Complete Prompt Gamma Analysis
  - Priority on items "potentially similar to 11589"
- Have requested DOE/RL guidance on ISP Destructive Analysis
- Deinventory
  - Plan for 2 year delay, but also be ready to ship in Spring 2007
  - Establish 9975 annual maintenance leak test capability
  - Proceed with certification of un-irradiated fuel package (HUFP)
- Detailed FY06 Surveillance Report Issued – HNF-31775

FY06 SRS 3013 CONTAINER SURVEILLANCE

Chip McClard

January 24, 2007

SRS FY06 NDE CONTAINERS

- 19 Random items
  - 1 Innocuous, 13 Pressure, 5 Pressure & Corrosion
- 3 Engineering Judgment items
  - FTIR shows HCL
  - Oldest container with > 10 grams water
  - Maximum calculated pressure
- 2 Additional items that were in facility for other measurements
  - 1 Pressure
  - 1 Pressure & Corrosion

SRS FY06 SURVEILLANCE RESULTS

- No anomalous conditions
- All surface smears less than 20 dpm/100 cm^3 alpha
- Visual observations good
  - No indications of corrosion on outer container
- Inner container gas pressures low
  - No snap-though of inner lid
- Prompt gamma – no change of bin designation
- Mass change less than 1.0 grams
- Full container radiographs - no detectable convenience or inner container failures

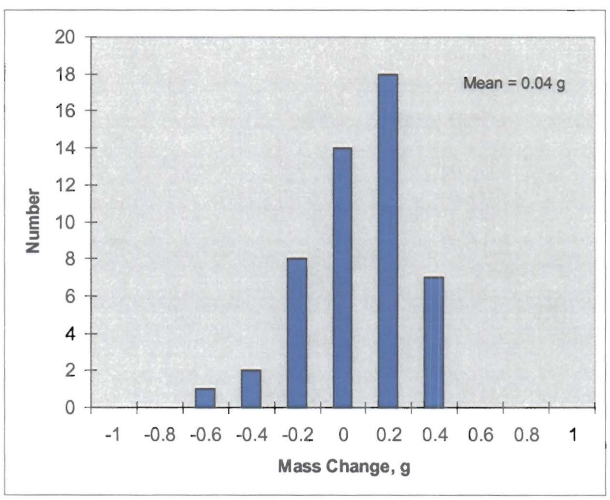
Pressure Verification

- For LES (FY05 and FY06) "manual" DR system used
  - Source with digital collector/camera
  - Engineer visually determined edge of lid and measured deflection using software aid
  - Used deflection and pressure correlation curves to assign conservative pressure
  - Adjusted $+2\sigma$ pressure to 412°F to compare to DSA
- Results indicate no inner lids have had snap-through
  - Pressures less than 25 psig
- KIS (FY07+) will have full DR system with deflection and pressure calculated by software (ADRIS)

Prompt Gamma

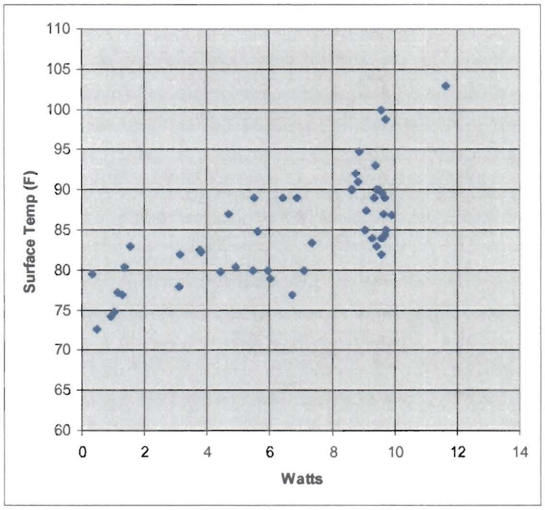
- Original RFETS PG total count time nominally 15 minutes
- SRS PG uses 60 minute live time
- Results indicate some changes to impurities (Joshua Narlesky to present more details)
- No changes to bin assignment

Mass Change FY05 and FY06 Data

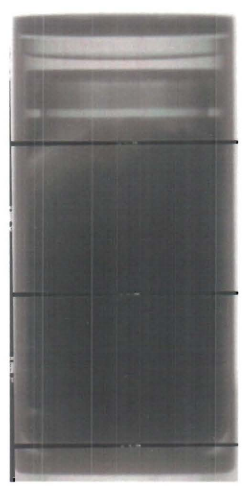


3013 Surface Temperature FY05 and FY06 Data

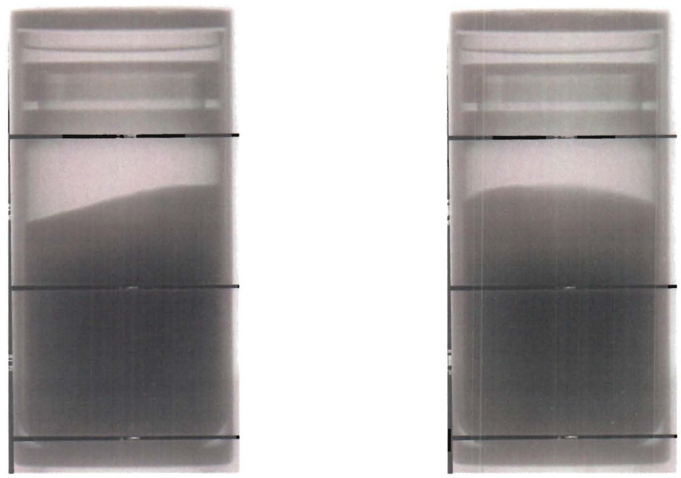
(Immediately after removing from 9975 PCV)



R611336 (Innocuous)

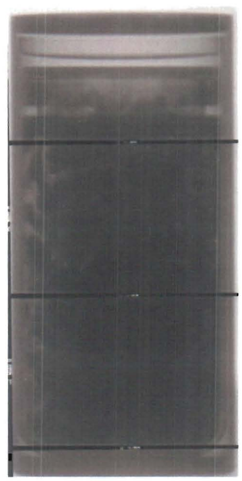


R600455 (Pressure)

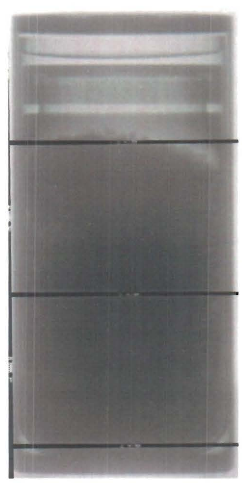


R610876 (Pressure)

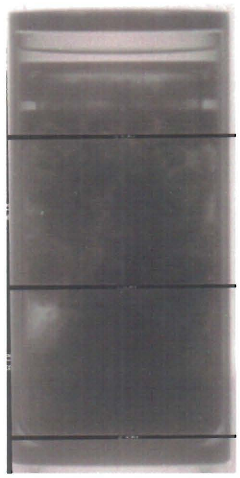
R610898 (P&C)

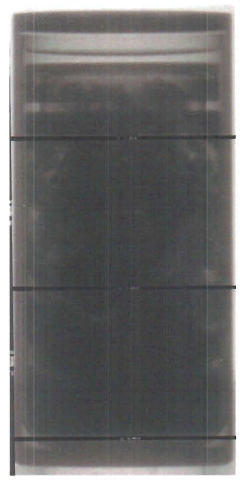


R611328 (P&C)



R610910 (P&C – HCl in FTIR)





CONCLUSION

- Two years of NDE complete
  - No unexpected results
  - Five year program to reach statistical confidence level
- In FY07, DE to begin and NDE to continue
  - Eleven year program to reach statistical confidence level for DE
- 36 Containers selected for FY07 surveillance
  - 13 NDE (Pressure) items at Hanford
  - 14 NDE (12 Pressure, 2 Innocuous) at SRS
  - 2 DE (Pressure) items at SRS (both had NDE in FY05)
  - 3 DE (Pressure and Corrosion) at SRS
  - 4 DE (Engineering Judgment) at SRS
    - 1 similar to ARF-223
    - 1 similar to C06032A
    - 2 similar to 011589A – defer until early FY08 because of flammability issue

DE Capability at SRS

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Kerry Dunn
SRS Pu Surveillance Program

January 23, 2007

Contributors: Chip McClard, Rich Koenig (K), Tina Stefek, Ron Livingston, Jon Duffey, Kurt Gardner, Zane Nelson, Art Jurgensen

K-Area Materials Storage (KAMS)

- 3013 containers stored within 9975 packages on palettes stacked up to 3 high
- 9975 packages must be transferred to KIS before they are opened
- Controls in place to minimize combustibles, avoid puncture of 9975, and pre-approve storage arrays
- IAEA packages within KAMS

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K-Area Interim Surveillance (KIS) Capability (FY07)

- Perform NDE of 3013 (primarily the same analysis in LES)
- Transfer 3013 to glovebox for DE prep
  - Puncture void between outer/inner can then through inner convenience can to capture gas samples
  - Section open each welded 3013 can (Section made at least ½" away from any closure or fabrication weld or HAZ)
  - Representative samples of oxide contents will be collected for analysis (moisture and chemistry)
- Cans, gas samples, and oxide samples will be sent to SRNL for analysis
- Oxide material repackaged in slip-lid can configuration for interim storage in 910B Vault
- Flowsheet analyses required for dissolution of material in HB-Line
- Excess oxide material and sample residues transferred from SRNL to HB-Line for processing

NOTE: CSSC Project will provide NDE, DE prep capabilities, 3013 can storage and re-packaging (welding) capability in FY09

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NDE Room

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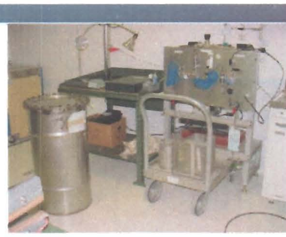
Equipment in NDE Room

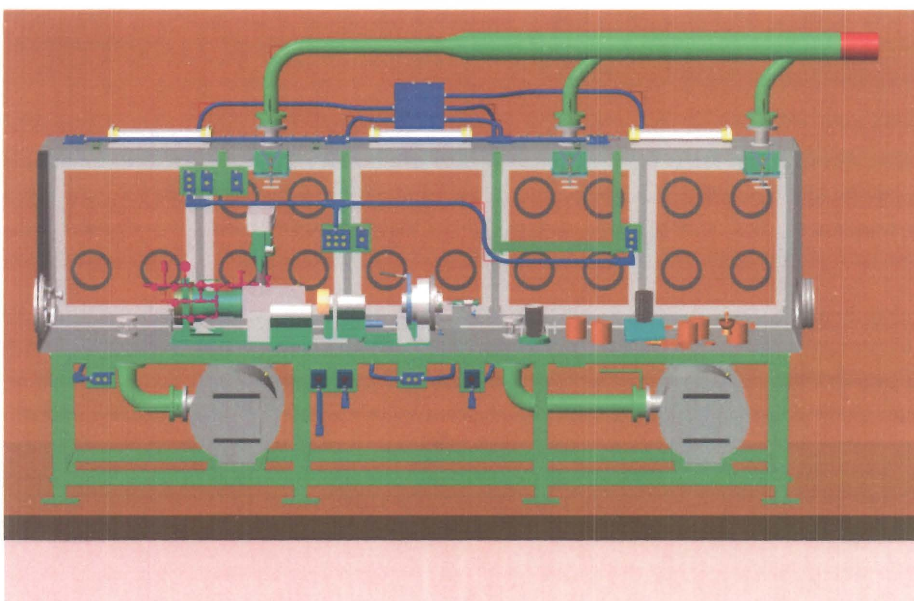


PCV/SCV Leak Test Unit

Digital Radiography

Prompt Gamma

Calorimeter (MC&A)



CPD in Glovebox

CPD Operation

- Pressure vessel that holds can horizontally
- Drill bit assembly to puncture containers
- Removable gas cylinders located adjacent to puncture location
- Single step closure mechanism
- Sample line purge cylinder for improved sample quality
- N_2 purge prior to puncture
- SS pressure gauge with data recorder

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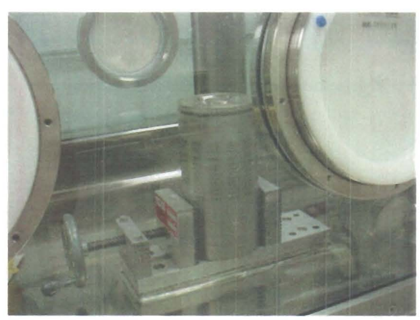
Series of KIS Gloveboxes

Can Cutter



Can Vise

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DPTE – Sample Transfer

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SRNL Activities

- Equipment Testing
  - Functionality of equipment to be used for the 3013 DE analyses was demonstrated in August
  - Included all equipment whether new or existing
- Demonstration of a 3013 DE
  - A mock-up demonstration of a 3013 DE was demonstrated in November
  - Included transfer of samples from KAC to SRNL, distribution of samples in SRNL, analyses, procedures, & training
- Developing Proficiency
  - In process of developing the proficiencies needed for 3013 DE

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SRNL Process Flow

- See attached file

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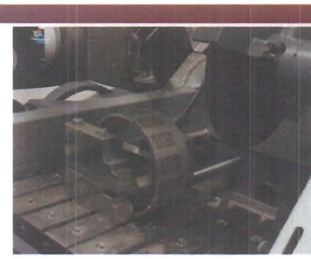
C-059 Glovebox



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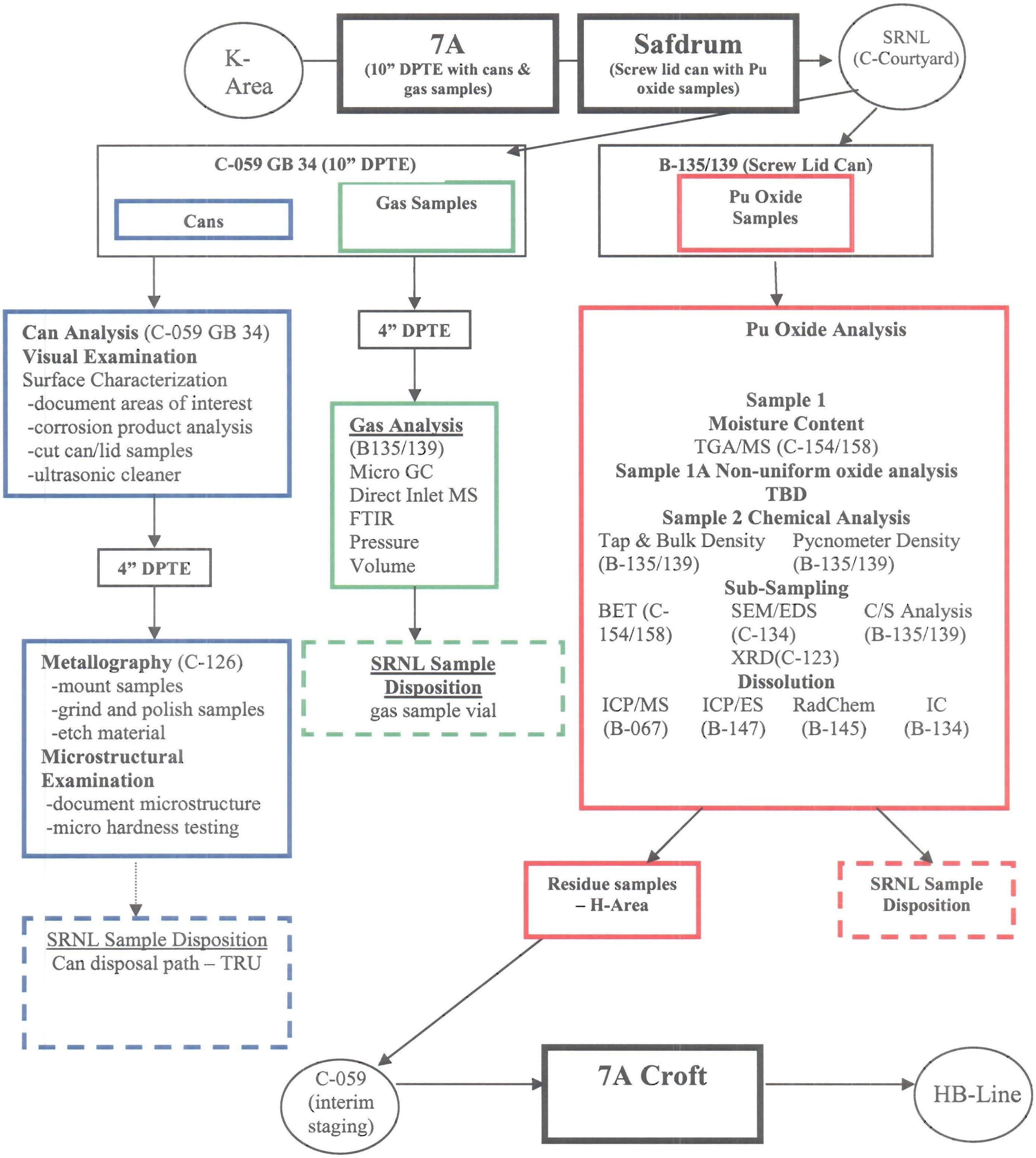
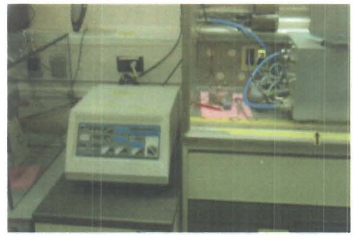
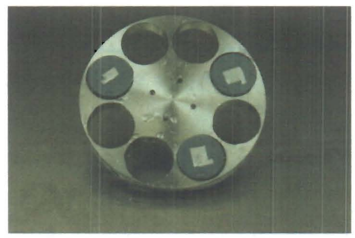
Process Flow Sheet for DE of 3013 Empty Containers, Headspace Gas, and Pu-Oxide



C-126 Equipment





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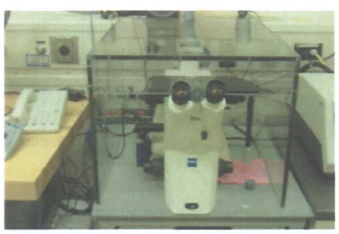
C-126 Equipment



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B-135/139 Room

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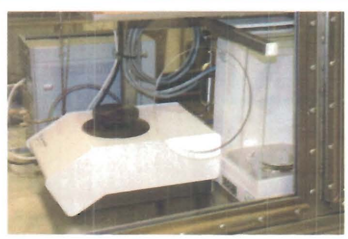
B 135/139 Equipment



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SRNL 3013 Destructive Analysis Status

- All analysis equipment in place, cold tested, and ready for hot operations
- La Cahlene double door transfer ports for transferring samples
- SRNL working to become proficient with sample transfers and analysis process

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SRNL DE Assumptions

- Each DE set include:
  - Three unpackaged 3013 container components (i.e. outer container, inner container and convenience can) for metallurgical evaluation,
  - Up to three headspace gas samples, and
  - Up to three plutonium oxide samples
- SRNL will have the capacity to perform one DE every two weeks and can complete up to 25 DE's per year. However, the normal operation is expected to be approximately 15 DE sets a year
- Plutonium oxide sample residues will be consolidated and sent to HB-Line

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KIS/SRNL Schedule

- SRNL facility ready for hot ops – developing proficiencies
- KIS Status
  - KIS is in final start-up testing mode
  - Procedures & training ongoing
  - MSA scheduled to start early March
  - DOE ORR scheduled for early May
  - DE Operations start scheduled for early June

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ISP Database - Completed/Future Plans at SRS

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We Put Science To Work

Tom Paul- ISP Modules
MIS Working Group Meeting

January 25, 2007

ISP Database

- Completed Modules
- Future Modules
- Pending Modifications

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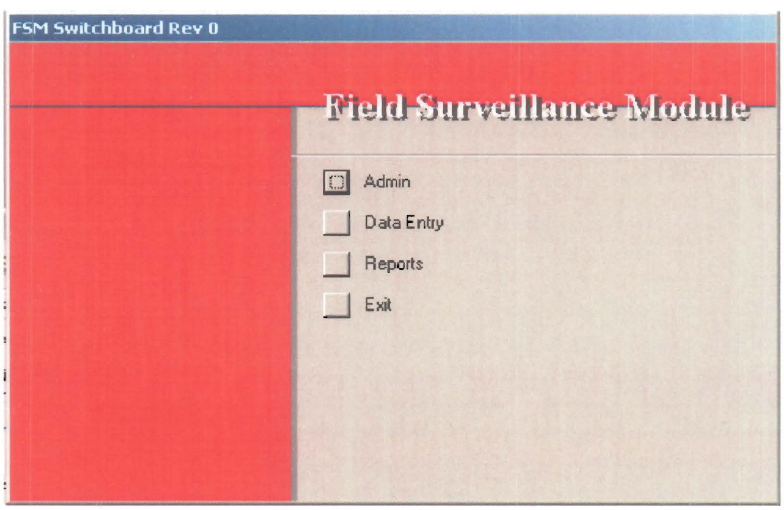
Completed Modules

- Field Surveillance Module (FSM)
- Shipping Container Surveillance Module (SCSM)
- Celotex® Module

Field Surveillance Module (FSM)



FSM (Continued)

- Data

  51 records entered to date

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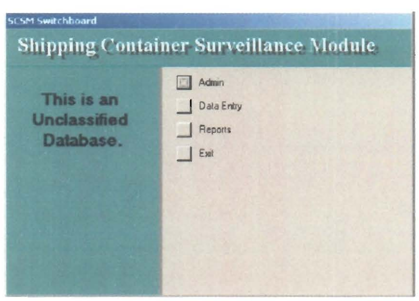
Shipping Container Surveillance Module (SCSM)



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SCSM (Continued)

- Data
  - 51 field records entered to date
  - 51 lab records entered to date
- Queries
  - 6 have been performed to date
- Reports
  - 7 have been created to date

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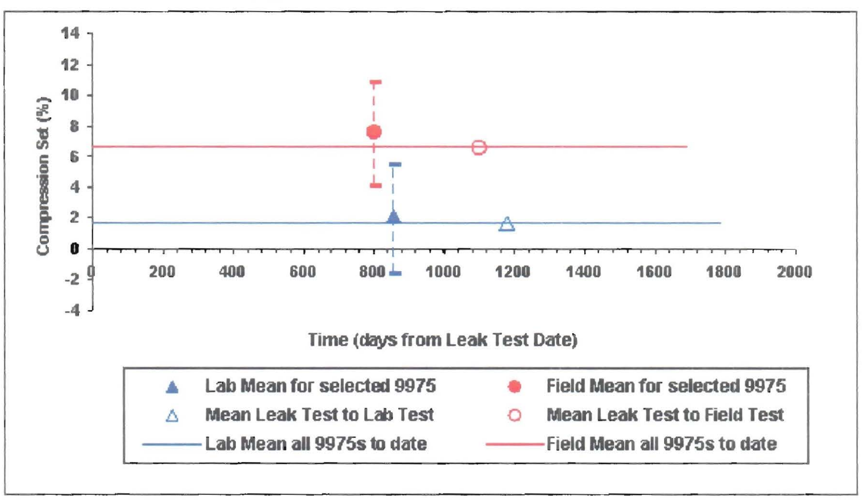
Graph Example One



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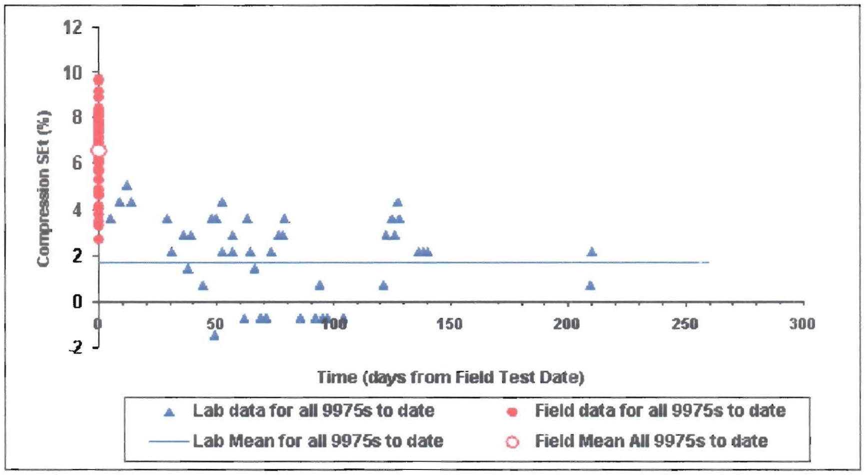
Graph Example Two



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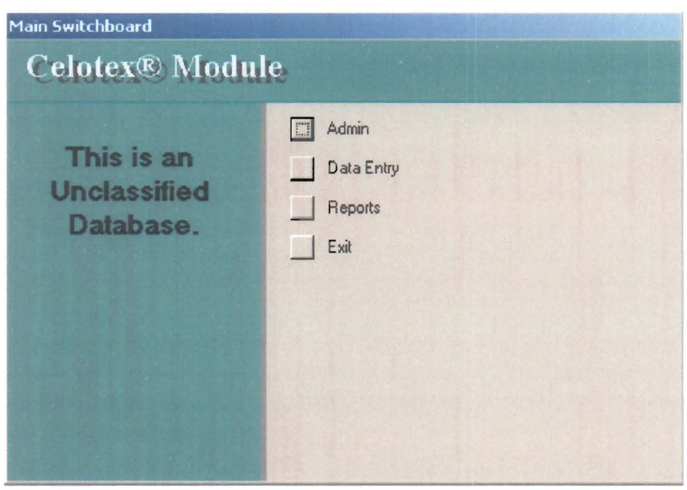
Celotex® Module



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Celotex® Module (Continued)

- Data

  3700 + records entered to date
- Queries

  None have been performed to date
- Reports

  None have been defined to date

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Future Modules

- 9975 Destructive Evaluation Module (4/07)
- SRNL 3013 Destructive Evaluation Module (6/07)
- KIS Destructive Evaluation Module (5/07)
- Corrosion Shelf Life Module (9/07)
- Life Extension Module (6/07)

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Pending Modifications

- Removal of 9975 field surveillance data from the FSM
- Incorporation of 9975 field surveillance data collection functionality into the SCSM
- Modification of the ISP database to provide users access to surveillance data

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ISP Database

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We Put Science To Work

Buzzy Cheadle
MIS Working Group Meeting

January 2007

ISP Database

- January 17, 2007 Release 3.1 Ready
- Deploy to SRS and Other Sites as Requested
- Data Concerns with ISP Data

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ISP Database – Data Concerns

- Resolve data issues with FB-Line metal batching
- Finalize 3013 packaging data from Hanford and Livermore
- Resolve additional items as they are found
- Resolve FY06 data issues with Livermore surveillance

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ISP Database – Baseline

- Decision to Baseline after FY05 and FY06 Surveillance Data Added to Database
- Only Approved Copy for Data Entry, Queries, etc.
- Working Databases are Not Official
- Surveillance Data Must be Sent to SRS from Offsite for Entering into Database

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ISP Database – Other Issues

- Begin Quarterly interactions with Los Alamos regarding ongoing developments (keep the ISP current with development efforts at the two sites)
- Creating ACREM is now possible at SRS
- Integrate or develop a surveillance data interface in the ISP
- Long Term Storage Retention Date?

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ISP Database - SRS Completed/Ongoing Efforts

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Gary Friday- ISP Queries
MIS Working Group Meeting

January 25, 2007

ISP Database

- Queries
- 011589A Technical Report
- Pressure Calculations
- Software Requirements Documents

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ISP Database - Queries

- Status
- Source
- Duration
- Protocol

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ISP Database Query Protocol

- Case Number
- Date Received
- Customer
- Statement of the Problem
- Resolution
- Closure
- Status

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011589A Technical Report

- August 2006
- Authors
- Objective
- Conclusions
- WSRC-TR-2006-00236
- gary.friday@srnl.doe.gov

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Pressure Calculations

- Objective
- Status
- Schedule

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Software Requirement Documents

- Basis and Objective
- Status
- Software Requirements Documents
  - 9975 O-Ring Module
  - 9975 Celotex Module
  - 9975 Destructive Evaluation
  - 3013 Destructive Evaluation (SRNL)
  - 3013 Destructive Evaluation (K-Area)
  - Corrosion Measurement & Evaluation
  - Integration of ISP Modules

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LLNL 3013 FY06 Surveillance Effort

January 24th, 2007
MIS/ISP Steering Meeting
Savannah River Site

Karen Dodson
Stephen Mayhugh
Lawrence Livermore National Laboratory

Lawrence Livermore National Laboratory, P.O. Box 808, Livermore, CA 94551

UCRL-PRES-228065

MIS identified one LLNL 3013 containers for NDE surveillance in CY-06

- The single LLNL containers selected for NDE are L000172
- Surveillance of L000172 is completed with the exception of the electronic data transfer to SRS (this will be discussed while we are here)
- LLNL is following the guidance in UCRL-TR-208875, "*The Lawrence Livermore National Laboratory DOE-STD-3013 Surveillance Program for the Storage of Plutonium Packages*"
- *The data has not been transferred to SRS as yet*

PUPPS-05-001, "3013 Surveillance," will be implemented for reporting the NDE

- The data for the surveillance requirements in UCRL-TR-208875 are captured using a Surveillance Report.
- The Surveillance Report contains the following information:
  - Container ID
  - Date of Surveillance
  - Operator name
  - Visual inspection for cracking, lid bulging, lid ok, pitting, and discoloration
  - Contamination data
  - DOE-STD-3013 package weight with a balance ID
  - Radiograph evaluation for lid deflection and Pu-bearing material condition
  - Package surface and ambient temperatures at time of surveillance
  - Perform prompt gamma analysis and calorimetry of the container contents and compare to previous data to verify contents

Cy-06 Surveillance Results of 3013 L000172

- Physical inspection shows no container deterioation, deformation or external signs of corrosion
- No surface contamination was detected
- The weight change was negligible
- The radiography revealed the physical existance of the material, no bulging lid on the inner can, and the distance between the lids has not changed
- The prompt gamma and calorimetry data correlated with the initial data
- The surface temperature was elevated over ambient

UCRL-TR-208875-Rev-1 Plan for Destructive Evaluation

- **UCRL-TR-208875-Rev-1 The Lawrence Livermore National Laboratory DOE_STD-3013 Surveillance Program for the Storage of Plutonium Packages has a expectation to begin destructive evaluation of a single can per year at SRS when the route to SRS is opened for shipment, originally 2007**
- **The new 2030 Complex NNSA Strategic Plan proposes the transfer of all Cat I/II SNM to "somewhere" by 2014, the inventory expected to be produced at LLNL is 250-400 3013 cans, thus about four NDE surveillance cans per year**

LLNL Repackaging Effort

Presented by:

David Riley

Surveillance & Monitoring Annual Meeting

October 17, 2006

This work was performed under the auspices of the U.S. Department of Energy by the University of California Lawrence Livermore National Laboratory under Contract No. W-7405-Eng-48.

Lawrence Livermore National Laboratory, P.O. Box 808, Livermore, CA 94551

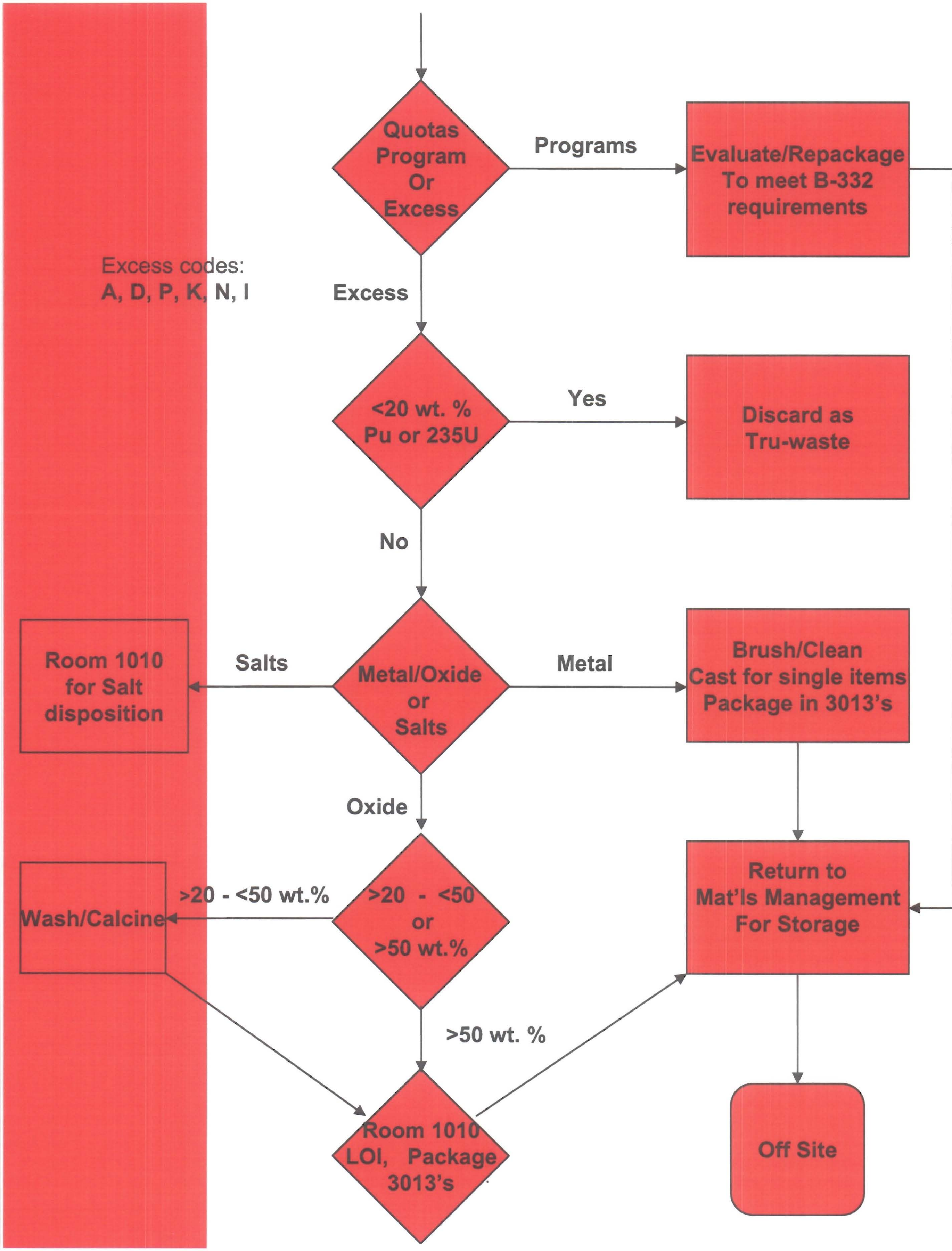
UCRL-PRES-228065

LLNL has been repackaging materials for a number of years for various reasons

- **Yellow label repackaging**
  - **The labeling requirements on containers changed**
    - The new labeling required more information about the material
  - **1700 items in 2001 had the old style label and needed to be visually inspected**
    - Most of these items have been opened and repackaged
      - We still have 3 items that require special processing
    - No packaging or potential packaging failures were found
- **5 year repack**
  - **We have made a commitment to repackage items older that 5 years**

A five year repack plan is being implemented

- Processing steps
  - Document current packaging
  - Insure proper characterization
  - Evaluate whether a package requires repackaging
  - If repackaging required, then open, inspect and repackage
  - Return to storage
- Five year evaluation and repackaging has been initiated
  - Oldest materials evaluated first
    - About 42 items older than 10 years.
      - Have processed 19 of them
      - 10 are standard that are being evaluated
    - About 275 items older than 5 years but less that 10 years
      - Have processed 11 of them
      - There are 21 standards that are being evaluated
    - Additional items will be added annually as they pass the 5 year mark



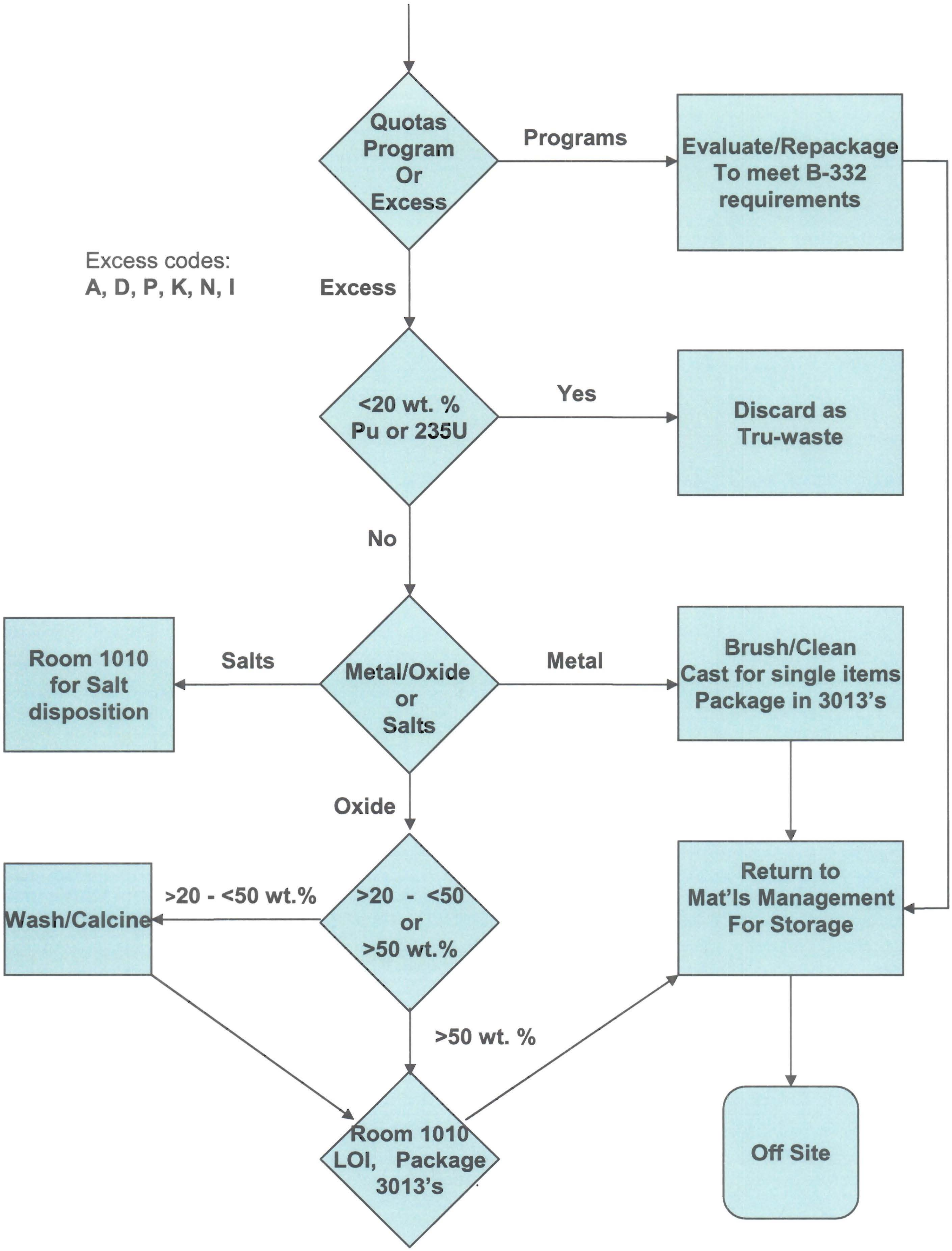
UCRL-PRES-228064

LLNL has been developing their own technical basis document for how non-DOE-STD-3013 material packages will be stored

- **Planning to use system similar to AWE for most packages**
  - **Screw top convenience container inside glovebox**
  - **Double containers outside of glovebox**
    - Double-crimp-sealed food pack cans
  - **Leak checking containers**
- **Pressure sealed containers (paint cans)**
  - **Use restraining clips**
- **Heavy duty, steel containers for items too large for food pack cans**
- **Overpacks**
  - **Screw top, heavy steel container (used by RFP in the pipe over pack)**
  - **DOT 10 gallon drum**
  - **Hagan can**

Packaging for items stored at LLNL is being identified

- Most Items
  - Oxides - Calcine before packaging
  - Screw top container
  - Bag out bag
  - Poultry bag
  - First Crimp Sealed can - helium leak check
  - Second Crimp Sealed can - helium leak check
- Pyrochemical Salts
  - One gallon paint can with retainers
  - Bag out bag
  - Poultry bag
  - Overpack in a DOT 10 Gallon, Hagen Can, or screw-lid steel can
- Multiple Items
  - Each item contained separately
    - Wrapped in aluminum foil or vial
  - Screw top container
  - Bag out bag
  - Poultry bag
  - First Crimp Sealed can - helium leak check
  - Second Crimp Sealed can - helium leak check


Quotas Program Or Excess
Programs
Evaluate/Repackage To meet B-332 requirements
Excess codes:
A, D, P, K, N, I
Excess
<20 wt. % Pu or 235U
Yes
Discard as Tru-waste
No
Room 1010 for Salt disposition
Salts
Metal/Oxide or Salts
Metal
Brush/Clean Cast for single items Package in 3013's
Oxide
Wash/Calcine
>20 - <50 wt.%
>20 - <50 or >50 wt.%
Return to Mat'ls Management For Storage
>50 wt. %
Room 1010 LOI, Package 3013's
Off Site

SRS CORROSION STUDIES

SRNL
SAVANNAH RIVER NATIONAL LABORATORY
We Put Science To Work

Jon Duffey, Ron Livingston, Bob Pierce, Mike Bronikowski,
Glen Kessinger, Phil Zapp (MTS)

Surveillance & Monitoring Annual Meeting
Savannah River Site

January 23, 2007

Outline

- Experimental approach
- Test apparatus
- PuO_2/salt mixture preparation
- Moisture uptake and sample loading
- Current status
- Path forward

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Experimental Approach

- Tests designed to evaluate effects of moisture, chloride salt composition and radiation dose on corrosion of test coupons
- Five test series with varying salt compositions
  - 21 samples have 0.5 wt% added moisture; three tests will be conducted at constant relative humidity of 60%
  - Three samples have increased alpha dose approximately 2.3X WG isotopic dose rate
- Test coupons
  - 304L and 316L stainless steel
  - Two flat coupons to evaluate pitting corrosion
  - Two tear drop coupons to evaluate stress corrosion cracking
- Heat to 70 ºC, monitor pressure, analyze headspace gas periodically
- Remove one sample from each set at approximately 3-month intervals to evaluate corrosion coupons

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Test Apparatus

- Test apparatus
  - Approximately 2"-diameter, 2.5"-tall stainless steel container
  - Sealed with a metal gasket using a chain clamp closure
  - Equipped with a pressure transducer and valve for acquiring gas samples
  - Glass inserts used to hold radioactive material in contact with test coupons
  - Vessels staged in controlled-temperature enclosures to maintain test temperature of 70 ºC

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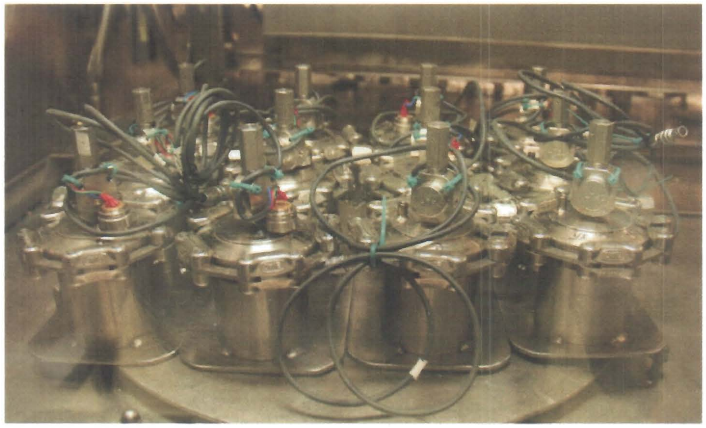
Test Vessels in Glovebox 49



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Glass Inserts with Oxide and Coupons

6

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Heated Enclosure

7

Heated Enclosure

8

PuO_2/Salt Mixture Preparation

- PuO_2 prepared by anion exchange, oxalate precipitation, and calcination to oxide at 950 ºC
- PuO_2 mixed with required amount of salt and heated to 850 ºC to form a homogeneous mixture
- PuO_2/salt mixtures were transferred into and sealed in glass jars while hot, then cooled and sealed in zip-loc bags to minimize moisture adsorption

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Corrosion Samples in Glovebox 44

Test Series	Sample ID	GB	Salt Content	Pu grams	Total grams	W/kg Pu
2	2A-1	44	10% NaCl/KCl	15.14	19.11	2.26
2	2A-2	44	10% NaCl/KCl	17.98	22.70	2.26
2	2A-3	44	10% NaCl/KCl	15.61	19.71	2.26
3	3A-1	44	10% ER Salt	16.29	20.57	2.26
3	3A-2	44	10% ER Salt	18.20	22.98	2.26
3	3A-3	44	10% ER Salt	19.67	24.84	2.26
3	3B-1	44	5% ER Salt	22.74	27.20	2.26
3	3B-2	44	5% ER Salt	21.29	25.47	2.26
3	3B-3	44	5% ER Salt	14.64	17.51	2.26
3	3C-1	44	2% ER Salt	18.65	21.62	2.26
3	3C-2	44	2% ER Salt	17.99	20.86	2.26
3	3C-3	44	2% ER Salt	19.98	23.16	2.26

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Corrosion Samples in Glovebox 49

Test Series	Sample ID	GB	Salt Content	Pu grams	Total grams	W/kg Pu
1	1A-1	49	0% Salt	18.63	21.17	2.26
1	1B-1	49	LANL Masterblend	19.45	30.70	2.26
1	1B-2	49	LANL Masterblend	21.73	34.30	2.26
4	4A-1	49	2% Ca ER Salt	17.88	20.73	2.26
4	4A-2	49	2% Ca ER Salt	17.55	20.35	2.26
4	4A-3	49	2% Ca ER Salt	16.71	19.37	2.26
4	4B-1	49	2% 11589 Salt	18.40	21.33	2.26
4	4B-2	49	2% 11589 Salt	16.37	18.99	2.26
4	4B-3	49	2% 11589 Salt	18.04	20.92	2.26
5	5A-1	49	5% ER Salt	18.55	22.19	5.09
5	5A-2	49	5% ER Salt	16.01	19.15	5.09
5	5A-3	49	5% ER Salt	20.31	24.30	5.09

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Moisture Uptake and Sample Loading

- PuO_2/salt mixture weighed into glass inserts in a dry (≤10% RH) helium atmosphere using a glovebag inside a radioactive glovebox
- Glovebag atmosphere humidified to promote moisture uptake; tracked by weight gain
- Samples loaded and sealed in test vessels in humidified helium atmosphere

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Current Status

- Twenty-four samples and two heater units staged in two gloveboxes at end of October 2006
- Startup of heaters has been delayed by ~ 3 months
  - Work stopped temporarily to evaluate Program funding in early November
  - AB issue of flammable gas generation in sealed containers emerged during stop-work; resolved in late December 2006
- As a result, samples have been sealed at ambient temperature for approximately 85 days
- Monitoring pressure for last 2 to 4 weeks

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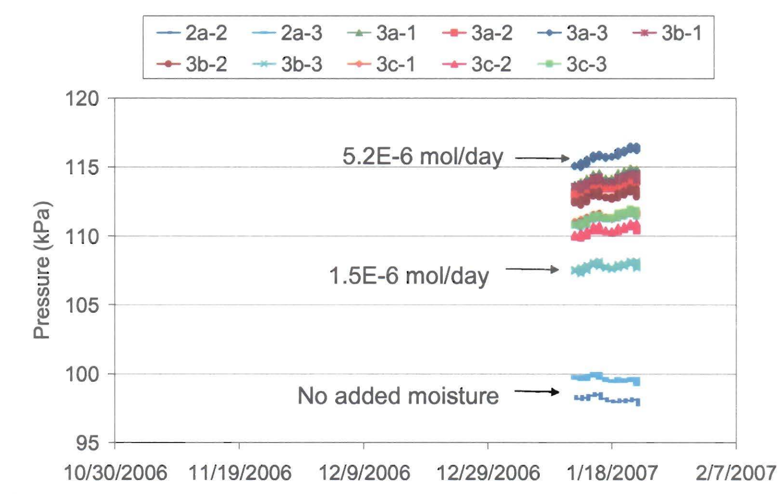
Test Series 2a (10% Na/KCl), 3a (10% ER Salt), 3b (5% ER Salt) and 3c (2% ER Salt)



14

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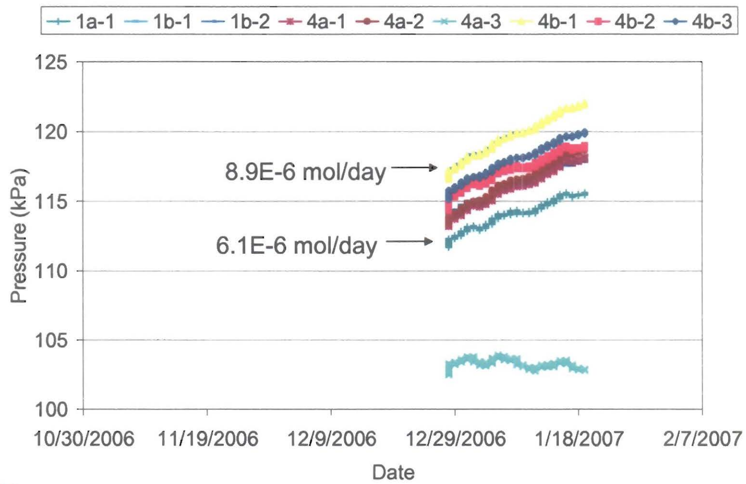
Test Series 1a (Pure PuO_2), 1b (LANL Masterblend), 4a (2% Alt Salt A), and 4b (2% Alt Salt B)



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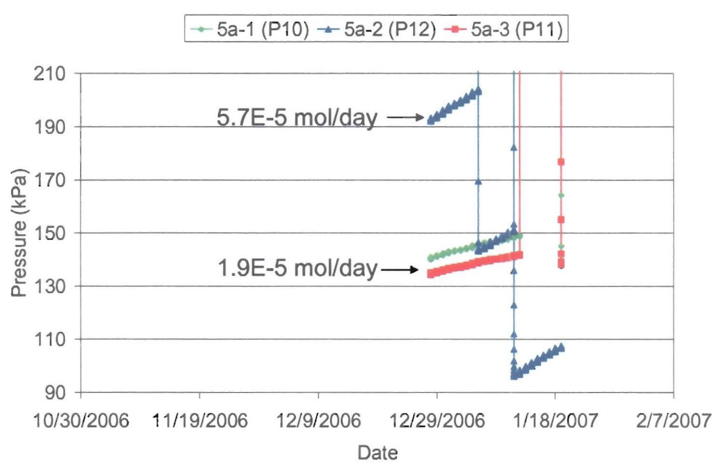
15

Test Series 5 (Increased Alpha Dose - 5% ER Salt)



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16

Path Forward

- Path forward discussed during teleconference with LANL team members last week
- Remove one sample from each set after storage at ambient temperature for approximately 90 days
- Heat some or all remaining samples to 70 ºC and remove second set after heating for 90 days
- Remove final set after time to be determined by Corrosion Evaluation team

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17

UNCLASSIFIED

Prioritization of LANL Nuclear Material for Repackaging and Stabilization Using Packaging Surveillance Information and MASS Database

Paul Smith
Jenifer Hoffman
Elizabeth Kelly
MIS Program Review
SRS, January, 2007

UNCLASSIFIED

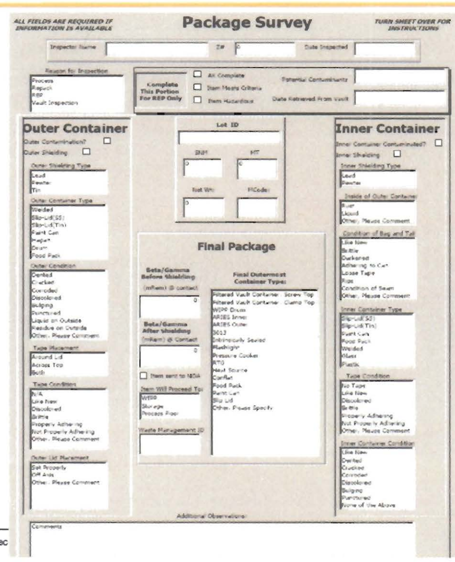
PURPOSE

- Enhance current risk prioritization method based on package surveillance information
- Determine if MASS data can be used for prioritization
- Compare package surveillance information to theoretical risk value

APPROACH

- Use package surveillance data to rank containers that have been repackaged
  - 685 containers ("learn" data)
  - Classify into "bad" containers and all the rest ("good ") based on ranking
- Use MASS information (IDES, AGE, NM, MT, LOTIDprefix) to predict "good" and "bad" containers for the learn data set
- Use MASS information to prioritize remaining containers (e.g. those needing repackaging)

Package Survey Form


ALL FIELDS ARE REQUIRED IF INFORMATION IS AVAILABLE

Package Survey

TURN SHEET OVER FOR INSTRUCTIONS

Inspector Name
Date Inspected

Reason for Inspection
Process
Repack
Vault Inspection

Complete This Portion For REP Only
All Complete
Item Meets Criteria
Item Hazardous
Potential Contaminants
Date Retrieved From Vault

Outer Container
Outer Contamination?
Outer Shielding

Outer Shielding Type
Lead
Pewter
Tin

Outer Container Type
Welded
Slip-Lid(SS)
Slip-Lid(Tin)
Paint Can
Drum
Food Pack

Outer Condition
Dented
Cracked
Corroded
Discolored
Bulging
Punctured
Liquid on Outside
Residue on Outside
Other, Please Comment

Tape Placement
Around Lid
Across Top
Both

Tape Condition
N/A
Like New
Discolored
Brittle
Properly Adhering
Not Properly Adhering
Other, Please Comment

Outer Lid Placement
Set Properly
Off Axis
Other, Please Comment

Lot ID
SNM
MT
Net Wt
MCode

Final Package
Beta/Gamma Before Shielding (mRem) @ contact
Beta/Gamma After Shielding (mRem) @ Contact
Item sent to NDA
Item Will Proceed To:
WIPP
Storage
Process Floor
Waste Management ID

Final Outermost Container Type:
Filtered Vault Container - Screw Top
Filtered Vault Container - Clamp Top
WIPP Drum
ARIES Inner
ARIES Outer
3013
Intrinsically Sealed
Pressure Cooker
RTG
Heat Source
Food Pack
Paint Can
Slip Lid
Other, Please Specify

Inner Container
Inner Container Contaminated?
Inner Shielding

Inner Shielding Type
Lead
Pewter

Inside of Outer Container
Liquid
Other, Please Comment

Condition of Bag and Tail
Like New
Brittle
Darkened
Adhering to Can
Loose Tape
Rips
Condition of Seam
Other, Please Comment

Inner Container Type
Slip-Lid(SS)
Slip-Lid(Tin)
Paint Can
Food Pack
Welded
Glass
Plastic

Tape Condition
No Tape
Like New
Discolored
Brittle
Properly Adhering
Not Properly Adhering
Other, Please Comment

Inner Container Condition
Like New
Dented
Cracked
Corroded
Discolored
Bulging
Punctured
None of the Above

Additional Observations
Comments

Challenge – How to Use Survey Data to Rank

- Over 1000 different configurations
  - Process bogged down when trying to go through all of these
- Solution: Multi-Attribute Utility Theory (MAUT)
  - Mathematical tool for ranking alternatives (e.g. different types of containers, packed in different ways, in different conditions)
    - Classic example – ranking cars based on price, reliability, safety ratings, fuel economy, and style
  - The technique makes a few elementary assumptions about
    - a) having an ability to make comparisons between two options, and
    - b) that preferences expressed are reasonably consistent (e.g. transitive and commutative laws apply)
  - Technique requires experts to reach consensus relative importance of attributes

MAUT PROCESS

- Experts developed preferences for individual attributes (e.g. condition of outer can, condition of lid, condition of inner can, condition of tape, etc.) (a two hour meeting)
  - Experts included processing technicians and staff members, inventory data experts, container experts, etc.

Example of Individual Attributes

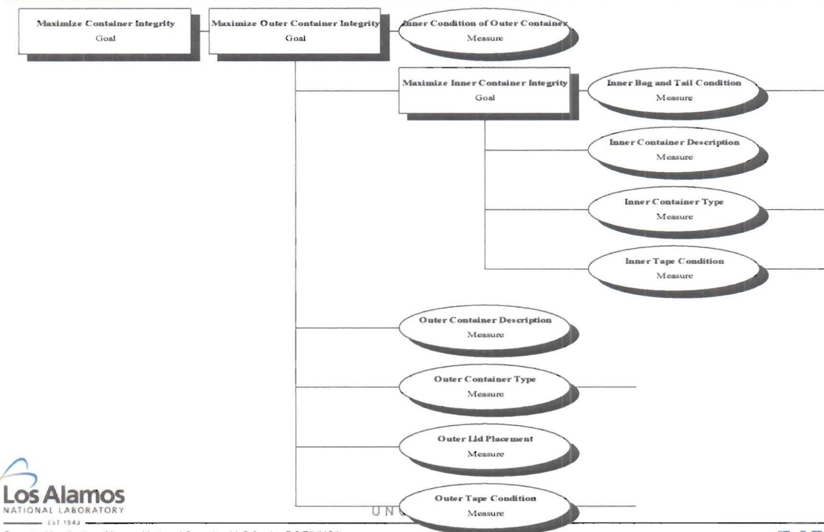
Measures	Sub Measure Categories	Sub Category Multipliers	Overall Measure Scale Summary
Inner Bag and Tail Condition		NA	0= least preferred alternative
	Bag Suppleness	10	Binary
	Bag Tape Condition	5	Binary
	Seam Condition	15	Binary
	Sep. from Can	10	Binary
Inner Condition of Outer Container		NA	Good (1), Rust (2), Liquid (3)
Inner Container Description		NA	No Container Issue (1), Discolored (2), Dented (3), Corroded (4), Cracked (5), Bulging (6)
Inner Container Type			0= least preferred alternative
	Food Pack	5	Binary
	Glass	0	Binary
	Paint Can	5	Binary
	Plastic	0	Binary
	Slip Lid SS	6	Binary
	Slip Lid Tin	5	Binary
	Welded	50	Binary
Inner Tape Condition		NA	0= least preferred alternative
	Inner Tape Adh. Proper	5	Binary
	Inner Tape Color	15	Binary
	Inner Tape Flexibility	10	Binary
Outer Container Description		NA	No Container Issue (1), Discolored (2), Dented (3), Corroded (4), Cracked (5), Bulging (6)
Outer Container Type		NA	0= least preferred alternative
	Food Pack	5	Binary
	Glass	0	Binary
	Paint Can	5	Binary
	Plastic	0	Binary
	Pressure Cooker	20	Binary
	Ring Drum-New	30	Binary
	Ring Drum-Old	0	Binary
	Slip Lid SS	6	Binary
	Slip Lid Tin	5	Binary
	SNMC	40	Binary
	Trashcan	0	Binary
	Welded	50	Binary
Outer Lid Placement		NA	0= least preferred alternative
Outer Tape Condition		NA	0= least preferred alternative
	Outer Tape Adh. Proper	5	Binary
	Outer Tape Color	15	Binary
	Outer Tape Flexibility	10	Binary
	Outer Tape Placement	15	Binary

be directly compared across
measures. The relative value of

Example of Individual Attributes (cont.)

Measures	Sub Measure Categories	Sub Category Multipliers	Overall Measure Scale Summary
Inner Bag and Tail Condition		NA	0= least preferred alternative
	Bag Suppleness	10	Binary
	Bag Tape Condition	5	Binary
	Seam Condition	15	Binary
	Sep. from Can	10	Binary
Inner Condition of Outer Container		NA	Good (1), Rust (2), Liquid (3)
Inner Container Description		NA	No Container Issue (1), Discolored (2), Dented (3), Corroded (4), Cracked (5), Bulging (6)
Inner Container Type			0= least preferred alternative
	Food Pack	5	Binary
	Glass	0	Binary

MODEL (based on first meeting)



MAUT PROCESS - Experts develop preferences for a subset of combined attributes

- EXAMPLE OF COMPARISON QUESTIONS
  Suppose you are presented with two outer containers. One has a lid which is improperly fit (type of container unknown) but has perfect outer tape. The second outer container (type unknown) has a perfectly fit lid, but outer tape that is improperly applied and in very poor condition. Which do you prefer? Why?
- *Eight such questions were answered in a one hour meeting*

MAUT RANKINGS

- Reviewed by experts for consistency with preferences
- Examples

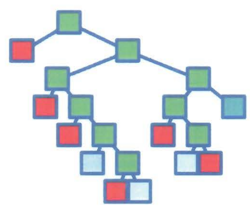
MAUT Rank	LOTID	MT	IDES	NM	AGE (yrs)	
3.60313901	RAG	83	K150	0.6	7.2	
3.10916963	MOX	52	C212	2877	11.3	
2.73333849	RAGP	83	K150	11.9	9.3	"bad"
2.73333849	RAG	83	K150	0.2	7.6	
2.73333849	COM	83	K00W	23.8	11.3	
4.45330138	FFR	54	R260	32	27.4	
14.6468223	CASLT	51	R092	53	8.6	
14.6468223	SLT	52	R422	1049	8.3	"good"
14.6468223	CASLT	52	R09A	7	7.8	
14.6468223	CR2	52	R092	29	14.0	
14.6468223	CASLT	52	R092	26	7.5	

CLASSIFICATION WITH MASS DATA

- "Learn" data set organized into "good" and "bad"
  - "Bad" an MAUT score less than 7 (54 bad)
  - "Good" everything else (616)
  - 15 items from original data not used because of lack of data
- Classification or Prediction Variables: NM, MT, IDES, AGE (*LOTID likely to be a good predictor but not enough items in LOTID prefix grouping*)
- Classification And Regression Trees (CART) (non parametric classification technique) applied to "learn" data

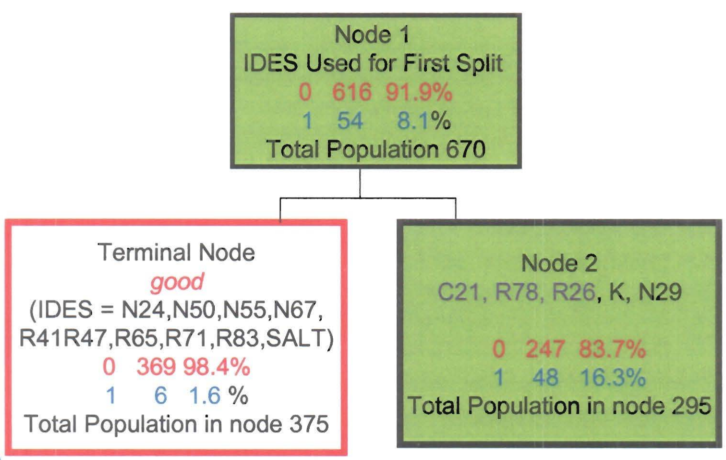
CART

- CART, using predictor variable information, splits data into nodes in a tree using an algorithm that tries maximizes the number of "good" items in a "good" node and "bad" items in a bad node under the constraint of minimizing the cost of misclassifications (*good terminal node*-red, *bad terminal node*-bluish, green-not a terminal node)



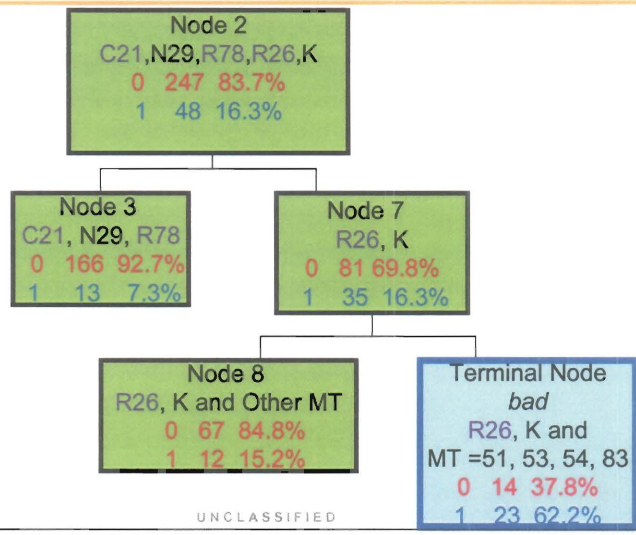
Tree Topology

CART: first split of the "learn" data set (0="good", 1="bad")

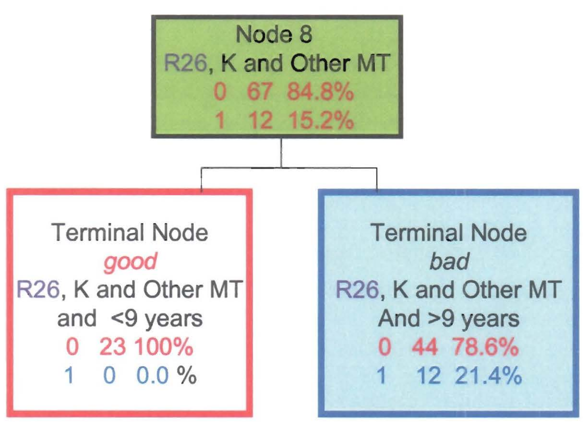


NOTE: C21, R78, R26 identified as high risk in previous studies

CART –Further Splits



Split of Node 8



RESULTS

IDES most important variable, age next and material type (MT) third. If remove IDES, then MT most important. Nuclear material amount did not contribute to results (possibly because low NM containers dominate learn database)

Note: % of "bad" in learn population 8% (want to target subpopulations with greater percentage of "bad")

Highest Priority – IDES types, K and R26 in combination with material types 51, 53, 54, and 83 ("bad" 63%) (all K, 83's except one were bad and all K, RAG (LotID prefix) were bad)

Second Highest – IDES types K and R26 with the other material types and greater than 9 years old ("bad" 21%)

Third Highest – IDES types C21, N29, R78 with material types 51,52,54 and 56 that are greater than 10 years old ("bad" 9%), greater than 18 years ("bad" 11%)

NOTE: Data show that IDES alone is not as good as IDES in combination with age and MT for classification/prioritization.

Filter Residue (R26), Feed for Dunn, (FFR)

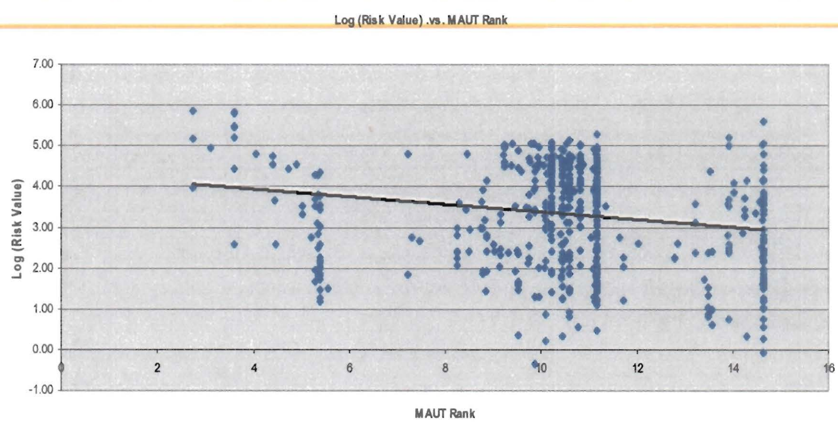
NEXT STEPS

- Score the remainder of the database

  Using CART
  - single tree

  Using Random Forest
  - hundreds of trees
  - voting algorithm
  - improved classification, but a "black box"

MAUT RANKING AND RISK VALUE



Some consistency, but a lot of spread:
The lowest risk items have high MAUT scores and the low MAUT scores have some of the highest risk items

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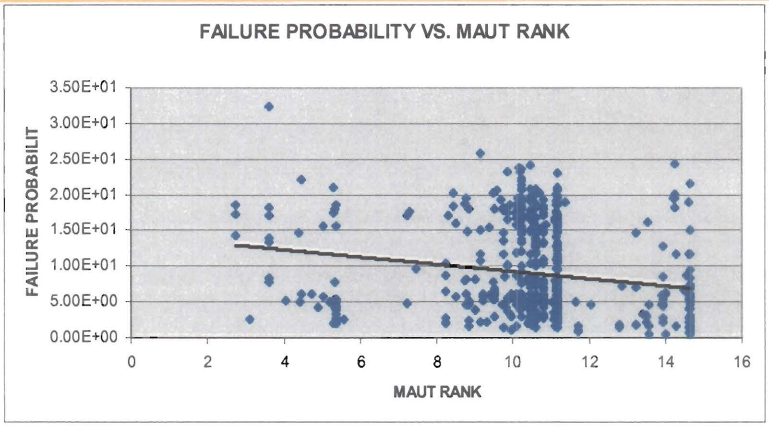
Failure Probability vs. MAUT Ranking



Some consistency, but a lot of spread: Highest failure prob has low rank and there is a trend of decreasing failure prob with increasing MAUT rank.

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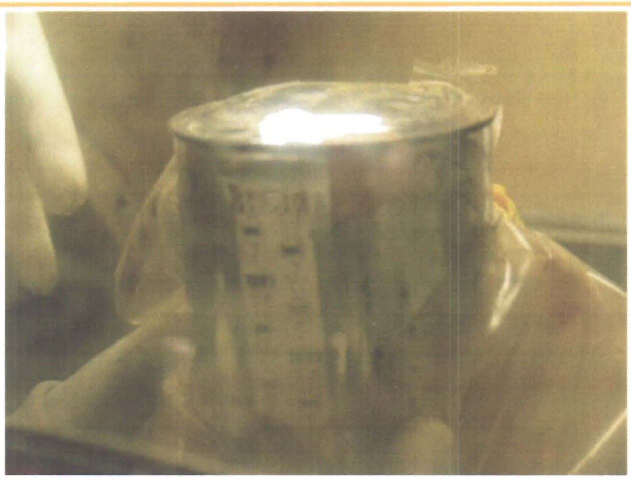
Bulging Container



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Prompt Gamma Analysis Update and Adsorption Correction

Joshua Narlesky

January 25, 2007

Scope

- Capability of Prompt Gamma Analysis
- Status
- NDE Assistance
- 011589A Issue & New Data
- Absorber Issue
- Software Updates

Prompt Gamma Capability

- Detects the following impurities when intimately mixed with Pu Oxide.
- Provides an estimate of concentration based on correlation with the normalized count rate.

Element	Approx. DL (wt%)
Al	0.45
Be	0.02
Cl	0.78
F	0.08
Mg	0.10
Na	0.004
P	0.33
K	0.98
Assume 60 minute count.	

Prompt Gamma Correlations

$$\log[X_i] = A \cdot \log(N_i) + B$$

Element	A	B	R^2	p	Concentration Range for MIS Samples
Al	0.478	4.823	0.684	4.3E-02	0.18 - 1.2 wt%
Be	0.813	4.527	0.924	9.2E-03	0.034 - 0.48 wt%
Cl	0.864	6.589	0.806	7.4E-05	0.45 - 7.7 wt%
F	1.029	5.870	0.911	6.3E-05	0.09 - 19 wt%
Mg	1.567	7.051	0.823	7.5E-06	0.002 - 31 wt%
Na	1.313	5.696	0.854	3.2E-10	0.003 - 2.4 wt%

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Status

- Currently: 3100 spectra completed
- Ongoing analysis of Hanford spectra
  - 69 new spectra in FY06
  - Items potentially similar to 011589A
- Ongoing NDE assistance
  - LLNL
    - FY05: 2 spectra—No significant change
    - FY06: 1 spectrum—0.4% Cl detected due to longer count
    - No bin change recommended
  - Hanford (PG only required if not completed)
    - No bin changes recommended
  - SRS
    - FY06: 22 spectra received
    - No bin changes recommended

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SRS NDE

- See spreadsheet

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LLNL NDE

- See spreadsheet

Hanford items “similar” to 011589

- See spreadsheet

Absorber Issue

- Estimates of concentration "not adding up"
  - Normalized count rates too high
  - Items with > 100% Cl or Mg??
- PG data suggests that absorbers were placed between the container and the detector
  - Detect n-gamma lines from "absorber" type material
  - Ratios of Pu-239 peaks not constant
- Hypothesis: Pb absorbers were used to reduce the dead time due to the 60 keV Am peak (and others). Can identify problem spectra by the ratio of Pu peak areas at 414 keV and 646 keV ($PR_{414/646}$)
  - Ratio < 30: appears 0.25" Pb used
  - Ratio < 10: appears 0.5" Pb used
- Impact: lower count rates for normalization peaks (414 keV and 662 keV) result in unreasonably high concentration estimates

Absorber Experiment

- Problem: Need justifiable method to identify the affected containers out of the 3100 that have PG spectra
  - Absorber use not recorded by sites
  - n-gamma lines not reliable; don't indicate absorber thickness
- Solution: Conduct experiment to determine $PR_{414/646}$ as a function of absorber thickness for MIS samples
  - Three samples selected: ARF-102-85-365 (strong chloride), MISNE4 and 053038 (numerous impurities)
  - Absorbers: 0.25", 0.5" 0.75" 1.0" Pb
  - Baseline count without absorbers

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Results of Absorber Experiment

- Results of ARF-365 and MISNE4 confirm hypothesized peak ratios.
- 053038 was labeled as “lead-lined”. Data matches average data if you assume a 1/8-in liner thickness

Absorber Thickness (in)	Pu Peak Ratio 414 keV/646 keV		
	ARF-365	MISNE4	Average
0	64	52	58
0.25	24	30	27
0.50	9.9	11.5	10.7
0.75	4.3	5.6	5.0
1.0	2.5	2.7	2.6

Curve equation: $R^2 = 0.9898$

$PR_{414/646} = 56.494e^{-3.1738x}$

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Corrections Applied (C)

Normalization Factor: $$N = \left(\frac{A_{414\,keV} / C_1}{BR_{414\,keV}} + \frac{A_{662\,keV} / C_2}{BR_{662\,keV}} \right) \cdot 10^{-6}$$

$$C_1 = \exp\left[\left(-\mu/\rho \right)_{Pb@414keV} \cdot (\rho x) \right]$$

$$C_2 = \exp\left[\left(-\mu/\rho \right)_{Pb@662keV} \cdot (\rho x) \right]$$

$$x = f\left(PR_{414/646} \right)$$

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Corrections Applied

Individual Peak Areas (i):

i = 2167 keV (Cl), 891 keV (F)…

$$A_{Corr,i} = A_i / \exp(-C_3 \cdot x)$$

$$C_3 = f(E_i)$$

Absorber thickness:

$$x = f\left(PR_{414/646} \right)$$

Corrected Data

Results for ARF-365

Absorber Thickness	Cl (wt%)		Mg (wt%)	
(in)	Corrected	Uncorrected	Corrected	Uncorrected
0	3.3	3.8	0.3	0.4
0.25	3.7	10.3	0.5	3.4
0.50	3.6	24.5	0.4	14.4
0.75	3.2	50.1	0.3	51.1
1.0	2.5	87.5	0.1	91.2
	Meas (by wet chem):3.8		Meas (by wet chem):0.55	

Impact on Database

- Containers affected ~35%, but only 6% had values of $PR_{414/646}$ less than 10.7
- Corrections reduced the estimated concentration
- Impact small at low concentration
- Impact on binning
  - Pressurization & Corrosion Criteria: >0.8% F
    - Before correction 166 > 0.8%
    - After correction 143 > 0.8%
  - Criteria for items like 011589: < 2% Cl
    - Before correction: 94 < 2%
    - After correction: 106 < 2%

Software Updates

- PGA v. 4.6 (January 2007) provided to SRS
- Calculates $PR_{414/646}$
  - Recommends a configuration (0.25" absorber, 0.5" absorber, etc.)
  - Performs absorber correction for given configuration)
- Provides concentration estimate in output file
- Includes a subroutine to create a summary table for the database
- Allows additional input format
- Converts PGR files to Ortec ASCII format (SPE)

FY07 Activities

- Analyze Hanford spectra; 783 oxides still require PG
- Analyze LANL spectra
- Assist sites with NDE
  - Training
  - Review of data
  - Data analysis
- Database activites

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DNFSB RECOMMENDATION 2000-1 MATERIAL STABILIZATION PROJECT UPDATE

W. A. Punjak, Obie Gillispie

Surveillance & Monitoring Annual Meeting
January 23-25, 200
Savannah River Site

00-1 "At a Glance"

- Implementation Plan (DOE/NNSA – DNFSB)
  - Stabilize ~1 metric ton of nuclear material
  - Stabilize = 3013, WIPP drum, site-standard container
  - Processes include burning/calcination, aqueous (NO3, Cl) recovery, and waste management
  - Milestones: WG(51-53), NWG(54-57, 42, 83), and the Recovery Evaluation Process (REP)
  - REP – items that might be discarded to WIPP
  - Scheduled completion October 2009

00-1 "At a Glance", con't.

- Project Execution Plan (LANL – DOE/NNSA)
  - More detailed than the implementation plan
  - Adheres to the implementation plan
  - 3013 packaging schedule by FY
  - Accounts for processing items previously stabilized through repackaging in site-standard containers
  - Scheduled completion end of FY12

Major Accomplishments

- Mid-point Milestones of IP Met
  - Stabilize 50% NWG (83 Kg) by 12/06
    - 118.5 Kg actually stabilized
  - Stabilize 50% WG (377 Kg) by 12/06
    - 383.8 Kg actually stabilized
  - Stabilize 50% through REP (124 Kg) by 12/06
    - 126.1 Kg actually processed

Challenges

- Unavailability of the RLWTF
  - Stabilization efforts moved from aqueous processing to repackaging
    - Items will have to be processed at a later date
    - Additional handling and cost
  - RLWTF expected to resume receipt of acid and caustic waste in February

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Challenges, con't.

- 12/05 vault contamination incident / fire suppression PISA
  - Stabilization efforts on hold for ~5 months in FY06
  - Items similar to failed item moved up in priority – all have been stabilized
  - Early gains still allowed IP milestones to be met

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Challenges, con't.

- Vessel stabilization / AB issues
- Stabilization of problematic items
  - Items requiring special handling (large, heavy, high dose, etc.)
  - All NWG (165 Kg) IP milestone: 12/07
- Resumption of 3013 packaging
  - Completion and approval of all 20 points

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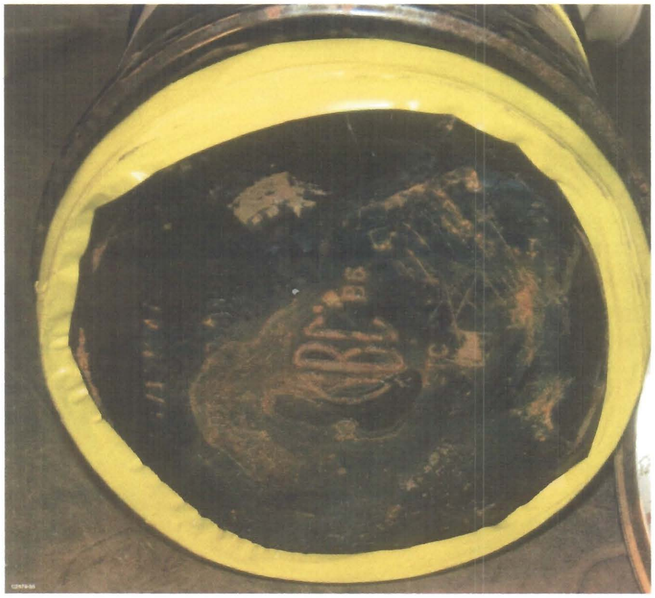

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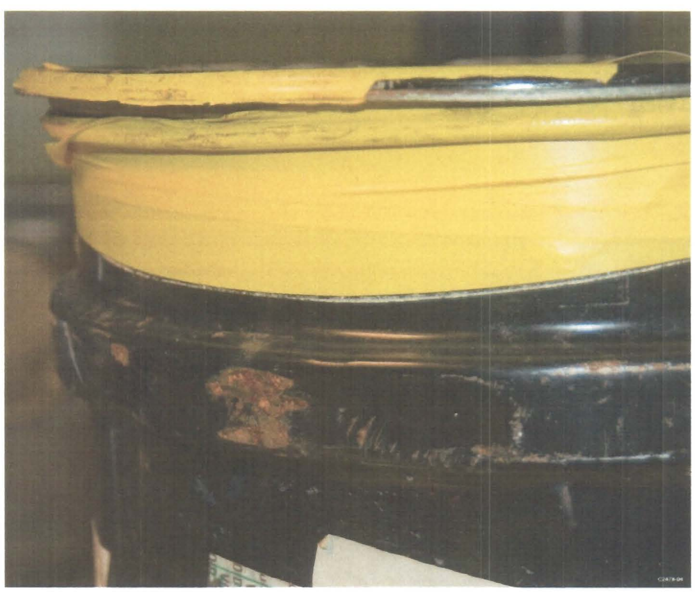

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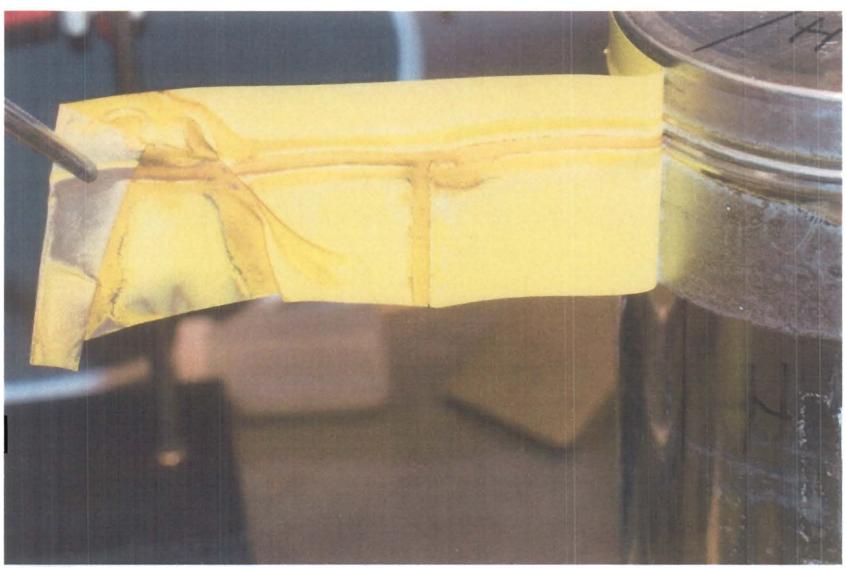
Tier 1 Items



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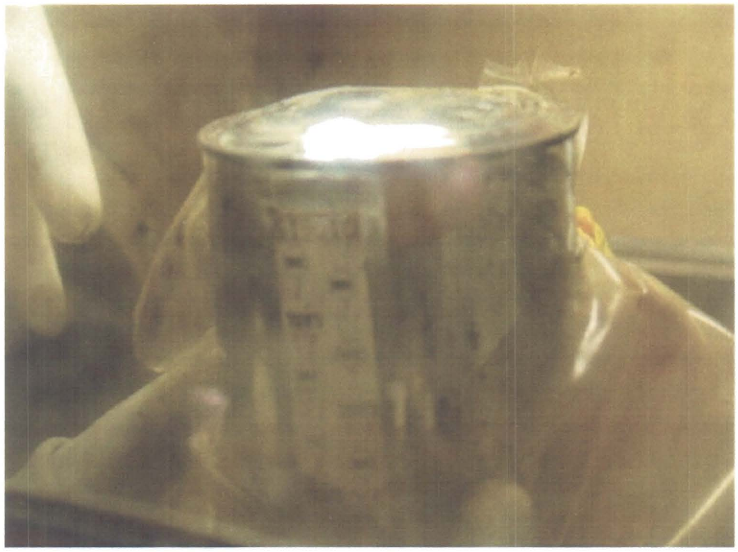


20 Points - Approved

- Qualification of welding technique/system (1)
  - Unofficially approved.
- Concurrence with welding procedures (5)
  - Unofficially approved.
- QA review of welder certifications and qualifications (6)
  - Unofficially approved.
- Container configuration different than BNFL (7)
  - Unofficially approved.

20 Points Update - Approved

- Oxidizing atmosphere (12)
  - Approved
- Sample handling (13)
  - Approved
- Moisture measurement (14)
  - Approved

20 Points Update – In Progress

- Overbatch (10)
  - Initial draft complete and submitted to SRS.
  - Meeting with SRS and WSMS personnel to review this week.
- Bed Temperature (11)
  - Testing in progress to allow operations at 1050°C
  - Prior testing allows operation at 1060°C. Document submitted to SRS for final review.
- MIS Representation (16)
  - List of items intended for 3013 stabilization and packaging is being compiled for submittal to SRS.
- Baseline Measurement (18)
  - Procedure developed and approved by LANL.
  - Initial draft to be complete for submittal to SRS by 2/28/07.

20 Points – In Progress

- Database (19)
  - Database initial development complete.
  - Database testing will begin in February 07.
  - Database procedures to be completed by March 07.
  - Initial draft to be complete by 2/28/07.
- Outer container test and qualification review and concurrence by SRS (3)
  - Awaiting official approval of Points 1, 5, 6, and 7

Summary

- Met project (IP) mid-point milestones
- Highest risk items being given priority with emphasis on releasing vault for non-respirator use
- Nearly ready to resume 3013 packaging
- Aqueous processing ready to resume as soon as RLWTF is available (2 weeks?)

Small-Scale Studies: Oxygen Generation and Corrosion

Ed Garcia, Max Martinez, Kirk Veirs, John Berg, Laura Worl
Los Alamos National Laboratory

Surveillance & Monitoring Annual Meeting
January 23-25, 2007
Savannah River Site

Questions to be Addressed

- Will a 11589 simulant generate oxygen?
- If so, what moisture levels are required?
- Is 11589 unique in generating oxygen?
- How does a PuO_2-$CaCl_2$ mixture compare to a 11589 simulant mixture and to an $MgCl_2$ mixture?
- Is there a relationship between corrosion-oxygen generation?

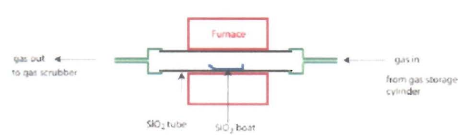
Small-Scale Samples

- SSR159A 5 wt. % $MgCl_2$
- SSR164 5 wt. % $CaCl_2$
- SSR165 2 wt. % 11589 Sim
- SSR166 1 wt. % 11589 Sim

Reagents
$MgCl_2$, $CaCl_2$ (99.9%) sealed in glass ampoules
NaCl, KCl reagent grade used as received
PuO_2 (PEOF1)

SSR159A Preparation

- PuO_2 + $MgCl_2$ ground together
- Heated 800 °C 1 hour Ar stream (0.2 slpm)



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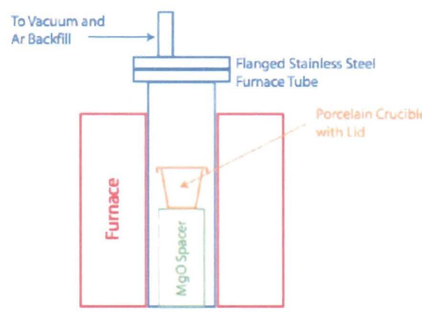
SSR164 Preparation

- PuO_2 + $CaCl_2$
- Heated 850 °C 1 hour static Ar



11589 Salt Simulant Preparation

ISE Analysis

Element	wt. %	wt. ratio
Mg	0.09	1.00
Ca	0.25	2.78
Na	0.26	2.89
K	0.33	3.67
Cl	1.02	

11589 Salt Simulant Preparation

Salt	wt. (g)	salt wt. ratio	metal wt. ratio
$MgCl_2$	0.2538	1.00	1.00
$CaCl_2$	0.7056	2.78	3.93
NaCl	0.7354	2.90	4.47
KCl	0.9314	3.67	7.54

Problem with 11589 ISE Analysis

Salt	Metal wt. %	wt. ratio Cl/M	wt. % Cl calc
$MgCl_2$	0.09	2.9	0.26
$CaCl_2$	0.25	1.8	0.45
NaCl	0.26	1.5	0.39
KCl	0.33	0.91	0.30

Calculated total Cl^- wt. % 1.4
Analyzed total Cl^- wt. % 1.0

40% discrepancy

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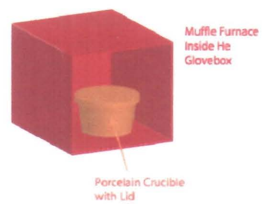
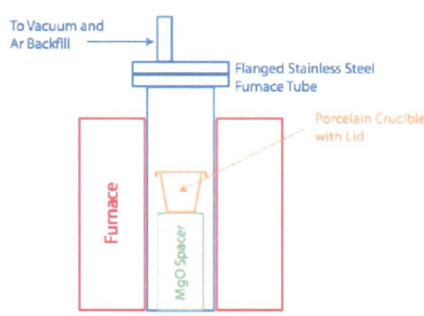
11589 Salt Simulant Preparation

Salts ground together in He atmosphere (3 ppm O_2)
Heated to 825 °C for 15 minutes



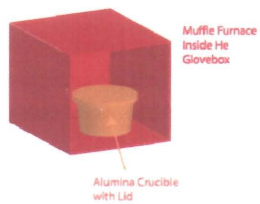
SSR165 Preparation

- PuO_2 + 11589 Salt Sim
- Heated 850 °C 1 hour static Ar



SSR166 Preparation

11589 Salt Sim ground with PuO_2 in He atmosphere
Heated to 825 °C for 15 minutes



Preparation Results

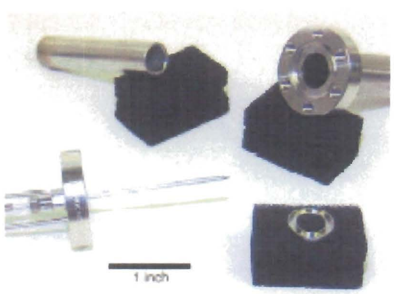
Experiment	Reactants	Salt wt. % as loaded	Salt wt. % post-prep from wt. loss	Salt wt. % post-prep ISE analysis
SSR159A	PuO_2 $MgCl_2$	5.11	4.29	4.19
SSR164	PuO_2 $CaCl_2$	4.98	NA	4.98
SSR165	PuO_2, 11589 Sim	2.30	NA	2.05
SSR166	PuO_2 11589 Sim	1.22	*	1.2

* A large weight loss was observed. Complete hydrolysis/oxidation of the $MgCl_2$ and $CaCl_2$ salt components would still not be enough to account for the weight loss.

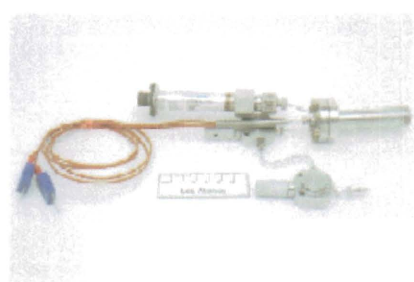
Hydration Procedure

- Powder placed on balance with humidified chamber
- Water uptake determined by weight gain
- At desired point sample sealed in small-scale container
- GC taken immediately after sample place on rack and after ~ 1 week
- Pressure monitored continuously
- Sample removed for next addition of water

Small-Scale Study Container

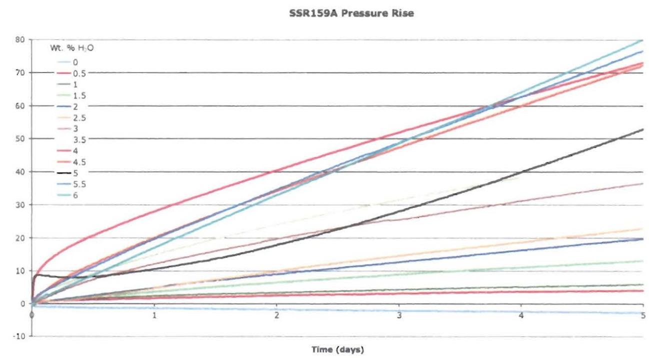


View of disassembled container for small-scale studies. Glass inner container used for all experiments except $MgCl_2$



Assembled container for studying pressure/gas generation of small samples. To the left can be seen thermocouples and pressure transducer

SSR159A ($MgCl_2$) Pressure



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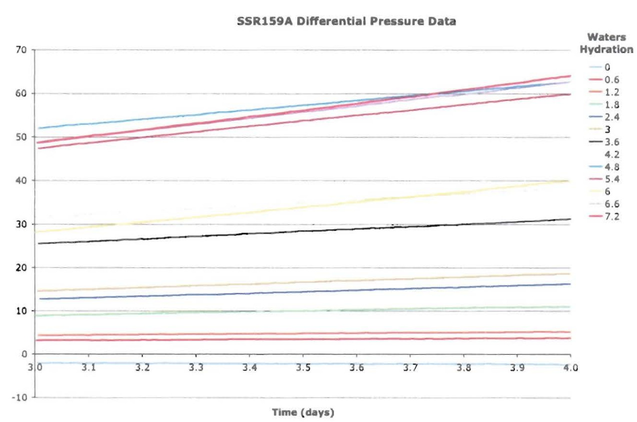
SSR159A ($MgCl_2$) Pressure



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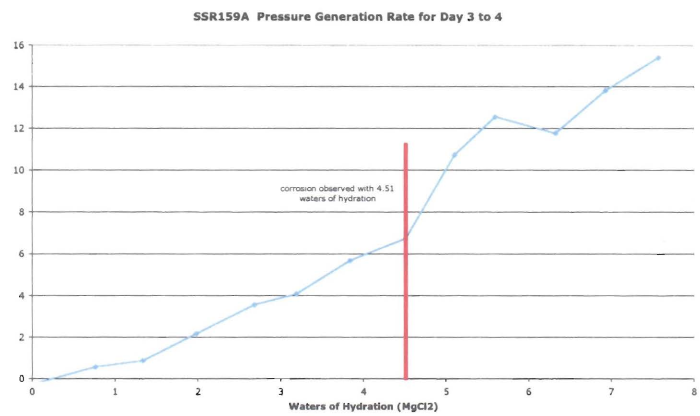
SSR159A ($MgCl_2$) Pressure Generation Rate



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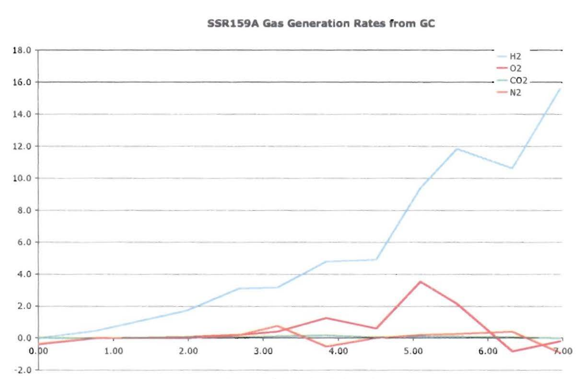
SSR159A ($MgCl_2$) GC Data



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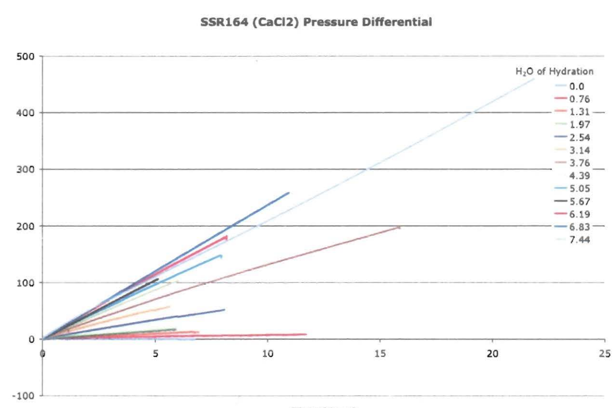
SSR159A Inner Container

ISE analysis yielded identical chloride content of powder at termination of experiment

SSR164 ($CaCl_2$) Pressure



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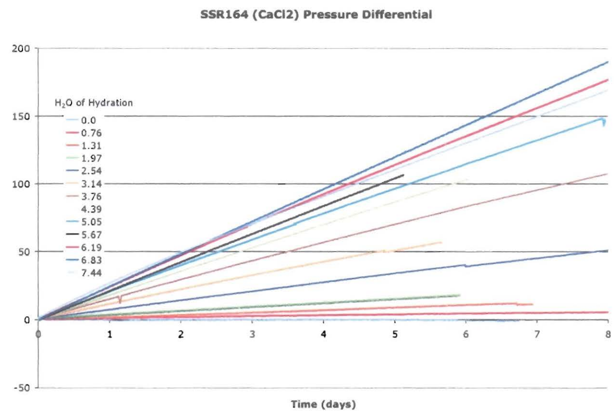
SSR164 ($CaCl_2$) Pressure



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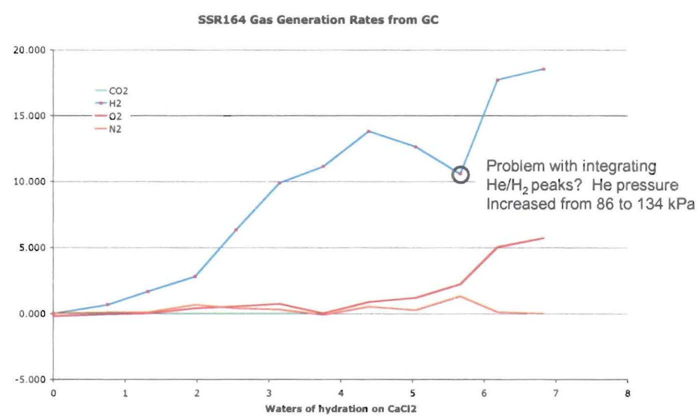
SSR164 ($CaCl_2$) GC Data



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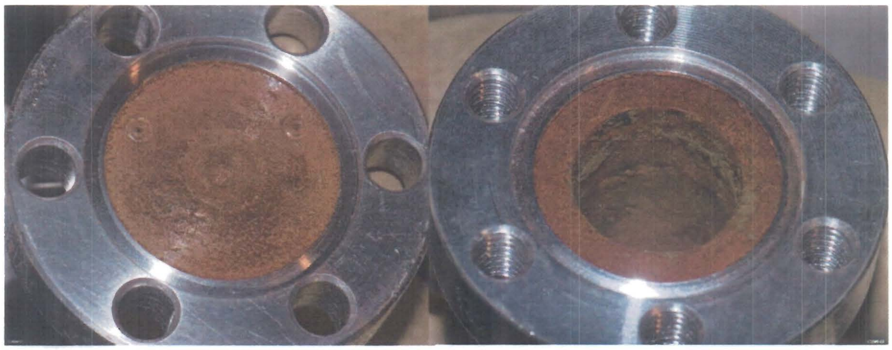
SSR164 ($CaCl_2$) Outer Container



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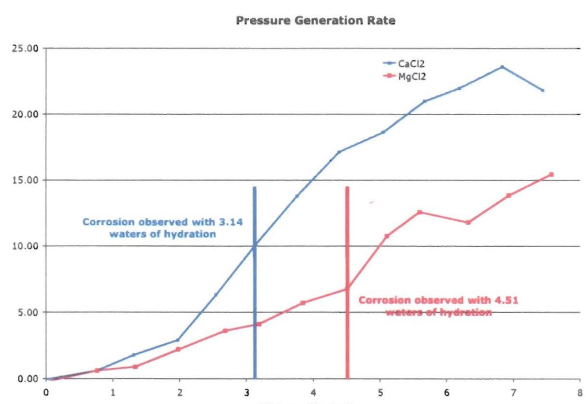
Pressure Generation $MgCl_2$ and $CaCl_2$



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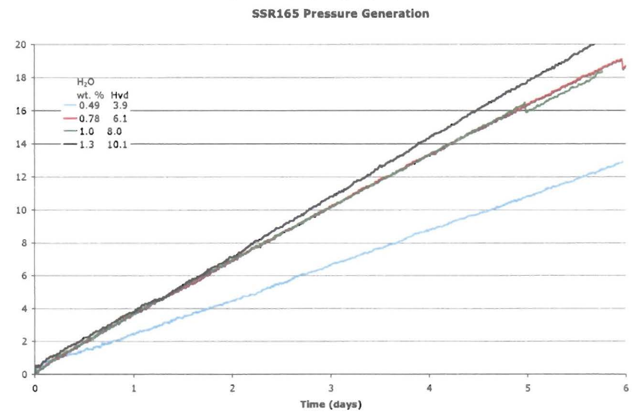
SSR165 (11589 Sim) Pressure Generation



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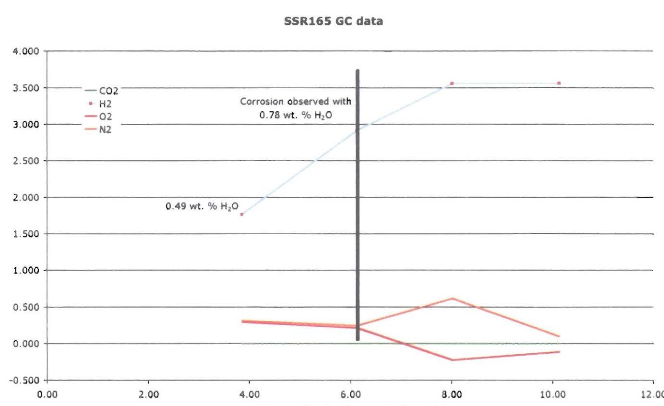
SSR165 (11589 Sim) GC Data



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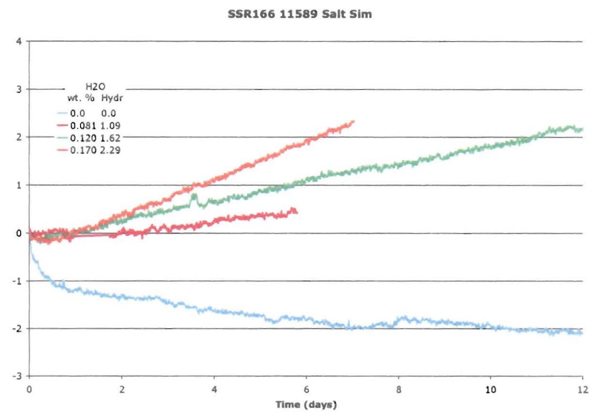
SSR166 (11589 Sim) Pressure



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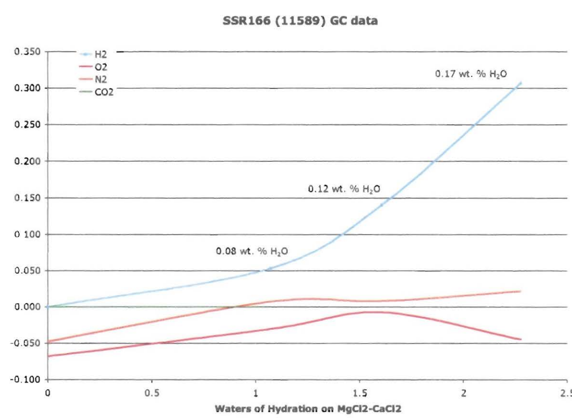
SSR166 (11589 Sim) GC Data



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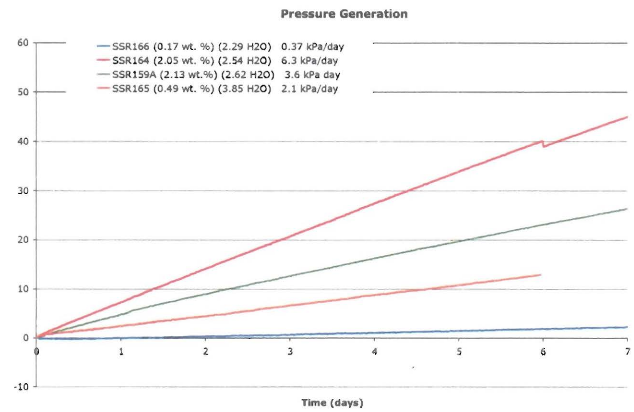
Pressure Generation 11589 Sim, $CaCl_2$, $MgCl_2$



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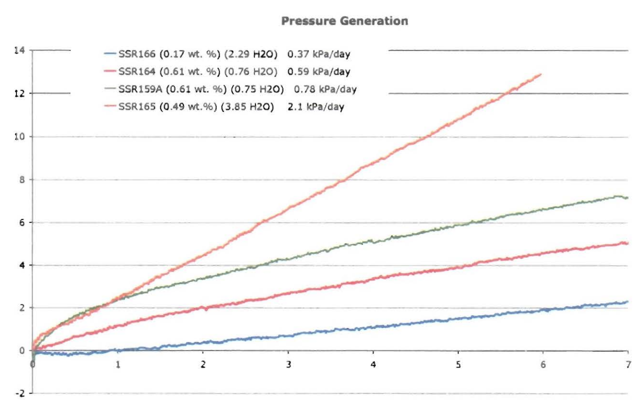
Pressure Generation 11589 Sim, $CaCl_2$, $MgCl_2$



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Summary

- A 11589 "simulant" will generate oxygen with moisture level of 0.5 wt. %
- $CaCl_2$ and $MgCl_2$ also generate oxygen
- $CaCl_2$ appears to be more active in terms of pressure generation-corrosion compared to $MgCl_2$
- Corrosion is observed before full hydration of the salts
- Oxygen generation rates seem to decrease once corrosion occurs
- For $MgCl_2$, no macroscopic loss of chloride from the powder is observed

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Conclusions

- Under varying conditions, oxygen is generated with all of the salts studied
- At very low moisture levels oxygen is consumed rather than generated
- Appears that oxygen generation starts at about 2 waters of hydration. This can be at or below 0.5 wt. % (SSR165)
- Corrosion can start before full hydration of the salt and seems to primarily dependent on waters of hydration (relative humidity)
- Pressure generation primarily dependent on absolute amount of water present (wt. %)

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Future Work

Finish 11589 Sim series with a 4 wt. % salt mixture to compare to the 1 and 2 wt. %

Repeat $MgCl_2$ and $CaCl_2$ with 2 wt. % salt

Small-Scale Surveillance and Oxygen Generation

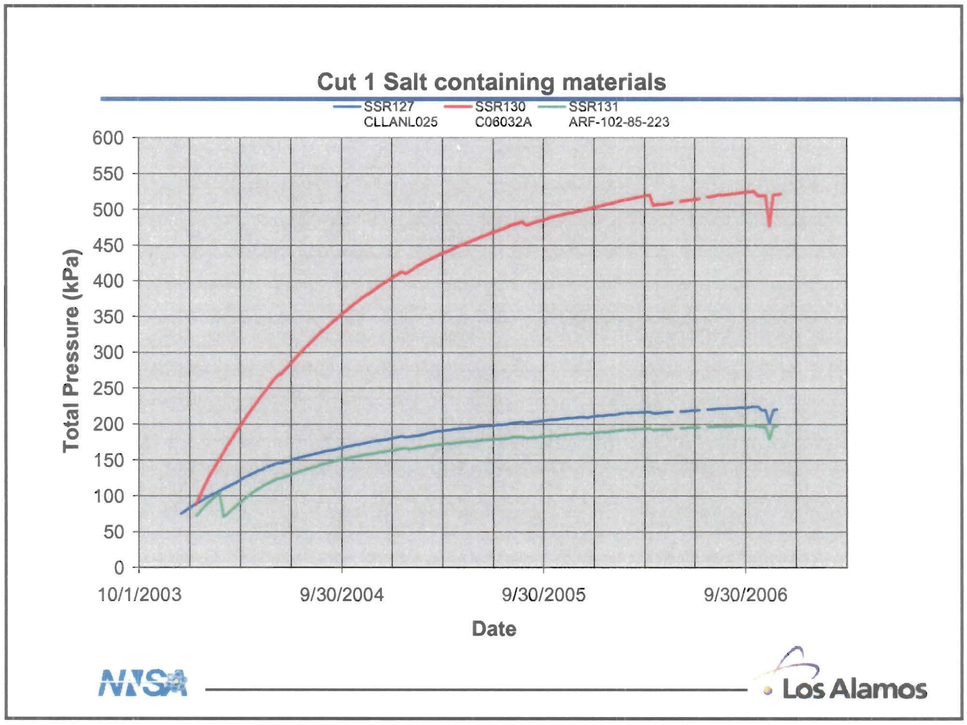
John Berg, Alex Carillo, Ed Garcia, Max Martinez, Dennis Padilla, Kirk Veirs, Laura Worl (PMT-1)
David Harradine, Rhonda McInroy (C-PCS)

7.501 Torr = 1 kPa
1 psi = 6.895 kPa

Surveillance and Monitoring Meeting
Jan. 23-25, 2007
Savannah River Site, SC

Outline

- Small-Scale Surveillance
  - Update pressure results.
  - H_2 gas generation rates.
  - Propose Small-Scale reactors to be removed.
  - Estimate relative humidity in small reactors.
  - Simple model for H_2 pressure evolution proposed to be implemented in the MIS database. Examples.


Cut 1 Salt containing materials
SSR127 CLLANL025
SSR130 C06032A
SSR131 ARF-102-85-223
Total Pressure (kPa)
0
50
100
150
200
250
300
350
400
450
500
550
600
10/1/2003
9/30/2004
9/30/2005
9/30/2006
Date
NNSA
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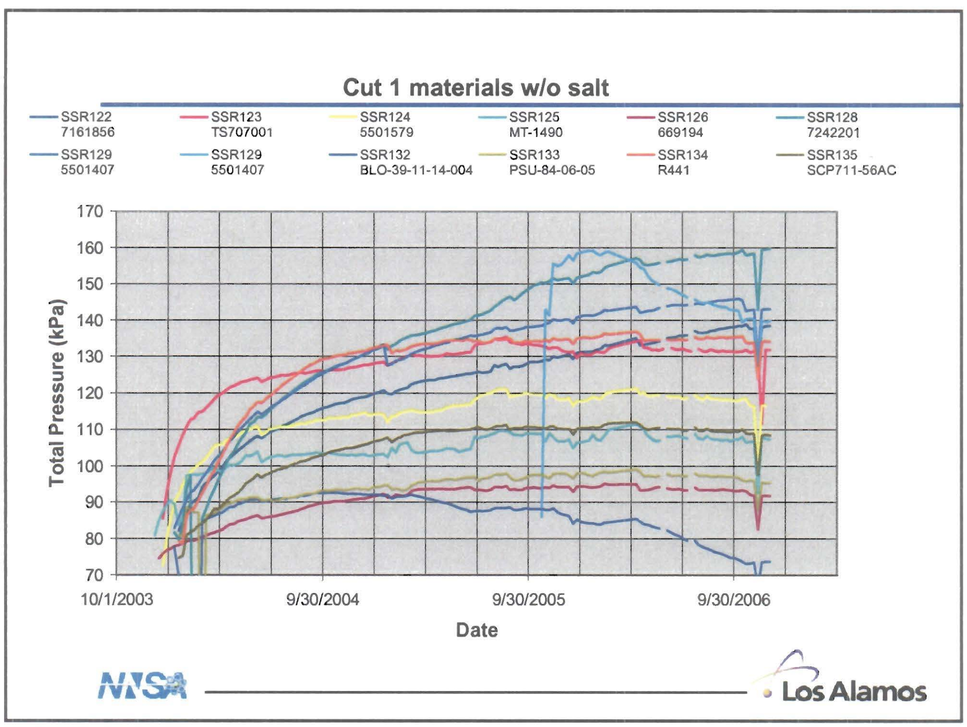
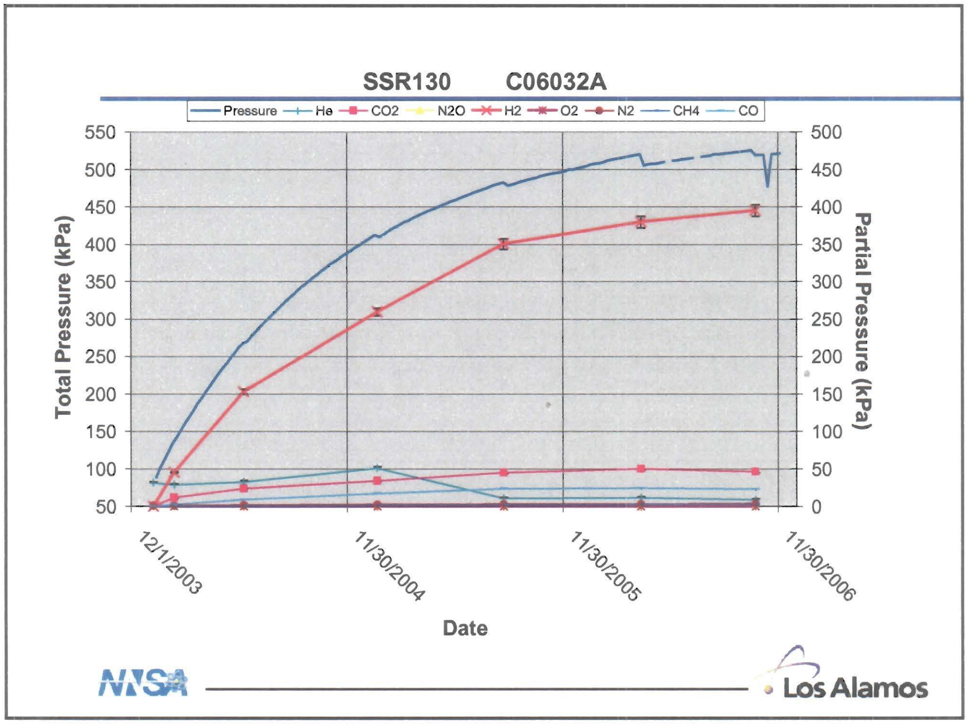
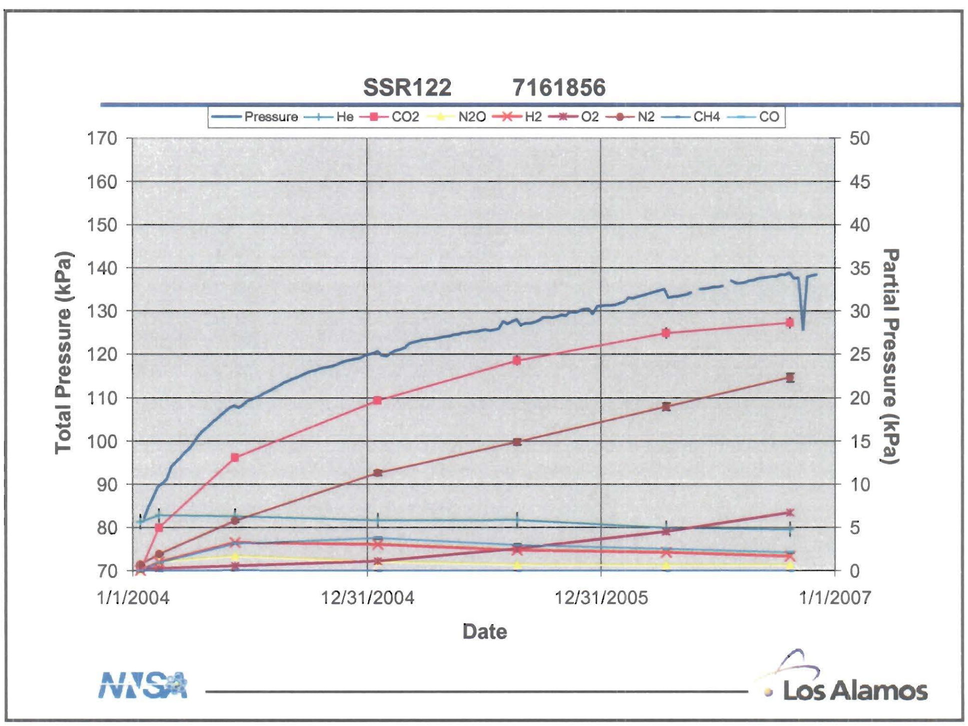

Cut 1 materials w/o salt
SSR122 7161856
SSR123 TS707001
SSR124 5501579
SSR125 MT-1490
SSR126 669194
SSR128 7242201
SSR129 5501407
SSR129 5501407
SSR132 BLO-39-11-14-004
SSR133 PSU-84-06-05
SSR134 R441
SSR135 SCP711-56AC
Total Pressure (kPa)
70
80
90
100
110
120
130
140
150
160
170
10/1/2003
9/30/2004
9/30/2005
9/30/2006
Date
NNSA
Los Alamos



SSR130 C06032A
Pressure He CO2 N2O H2 O2 N2 CH4 CO
Total Pressure (kPa)
Partial Pressure (kPa)
550
500
450
400
350
300
250
200
150
100
50
0
12/1/2003
11/30/2004
11/30/2005
11/30/2006
Date
NNSA
Los Alamos



SSR122 7161856
Pressure He CO2 N2O H2 O2 N2 CH4 CO
Total Pressure (kPa)
Partial Pressure (kPa)
170
160
150
140
130
120
110
100
90
80
70
50
45
40
35
30
25
20
15
10
5
0
1/1/2004
12/31/2004
12/31/2005
1/1/2007
Date
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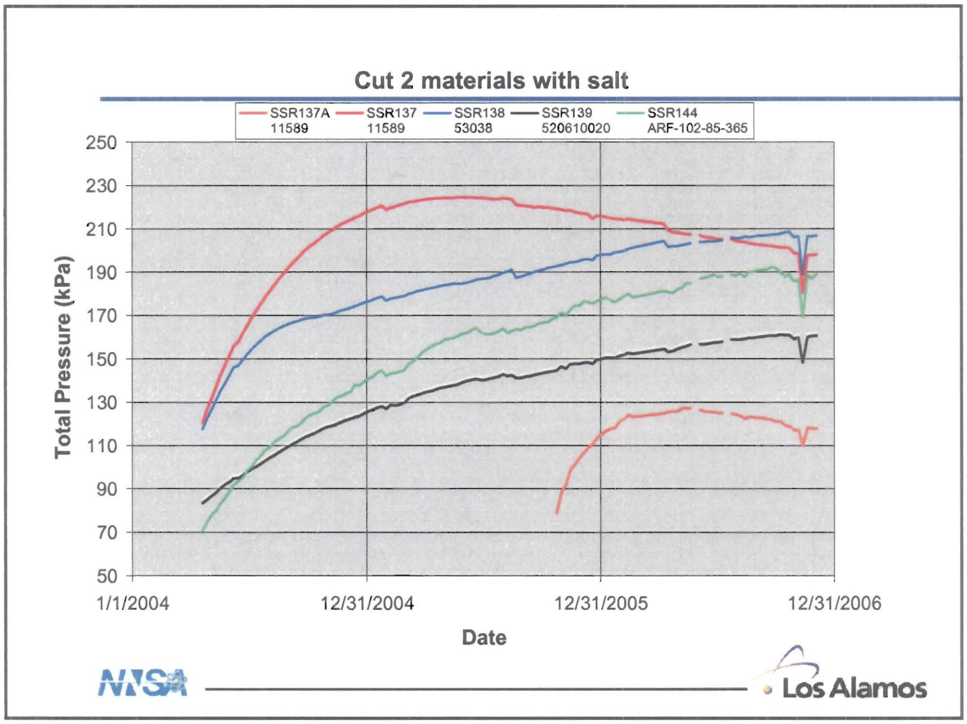
Cut 2 materials with salt
SSR137A 11589
SSR137 11589
SSR138 53038
SSR139 520610020
SSR144 ARF-102-85-365
Total Pressure (kPa)
250
230
210
190
170
150
130
110
90
70
50
1/1/2004
12/31/2004
12/31/2005
12/31/2006
Date
NNSA
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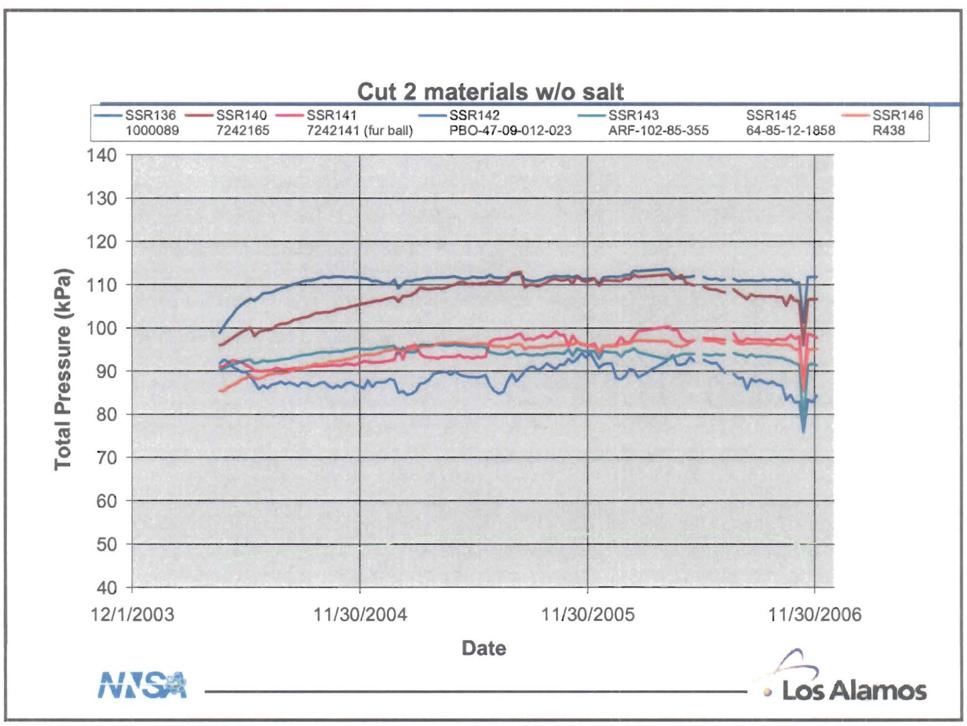
Cut 2 materials w/o salt
SSR136 1000089
SSR140 7242165
SSR141 7242141 (fur ball)
SSR142 PBO-47-09-012-023
SSR143 ARF-102-85-355
SSR145 64-85-12-1858
SSR146 R438
Total Pressure (kPa)
140
130
120
110
100
90
80
70
60
50
40
12/1/2003
11/30/2004
11/30/2005
11/30/2006
Date
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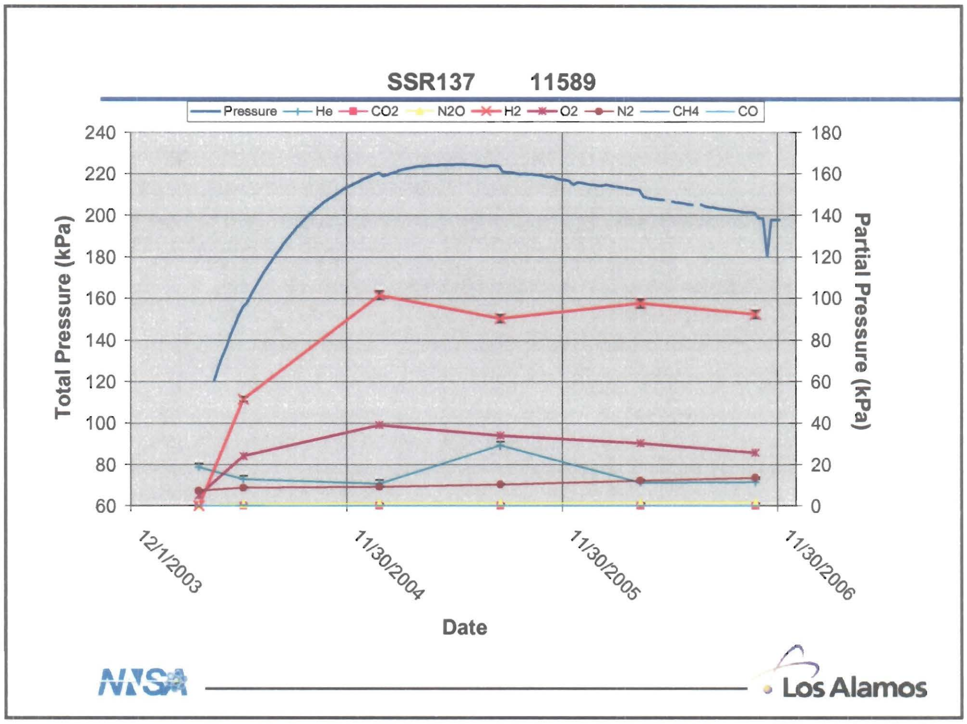
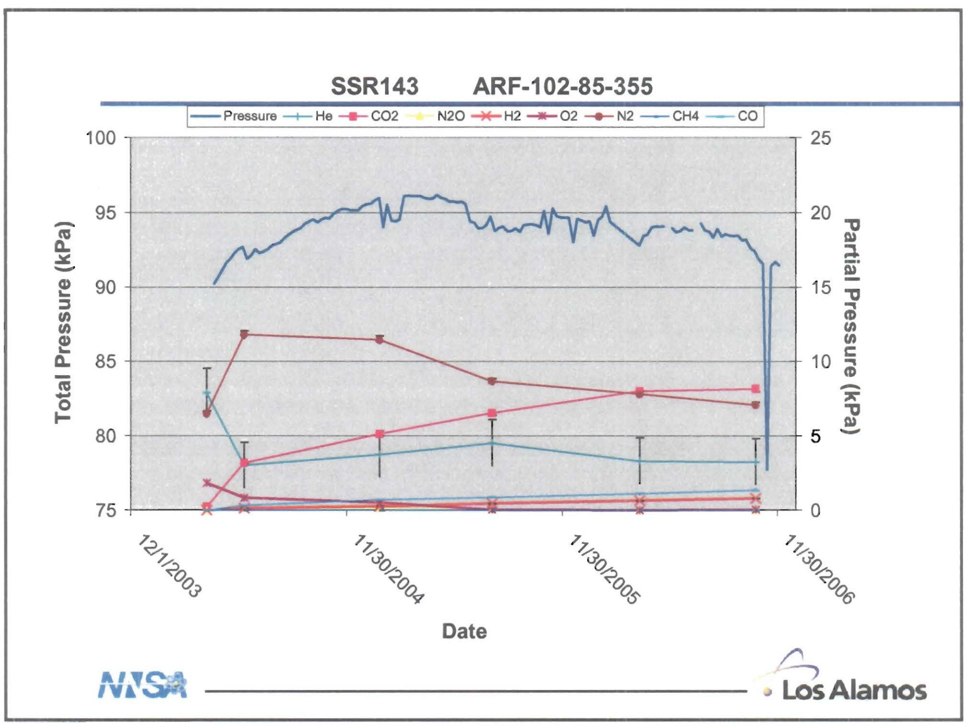
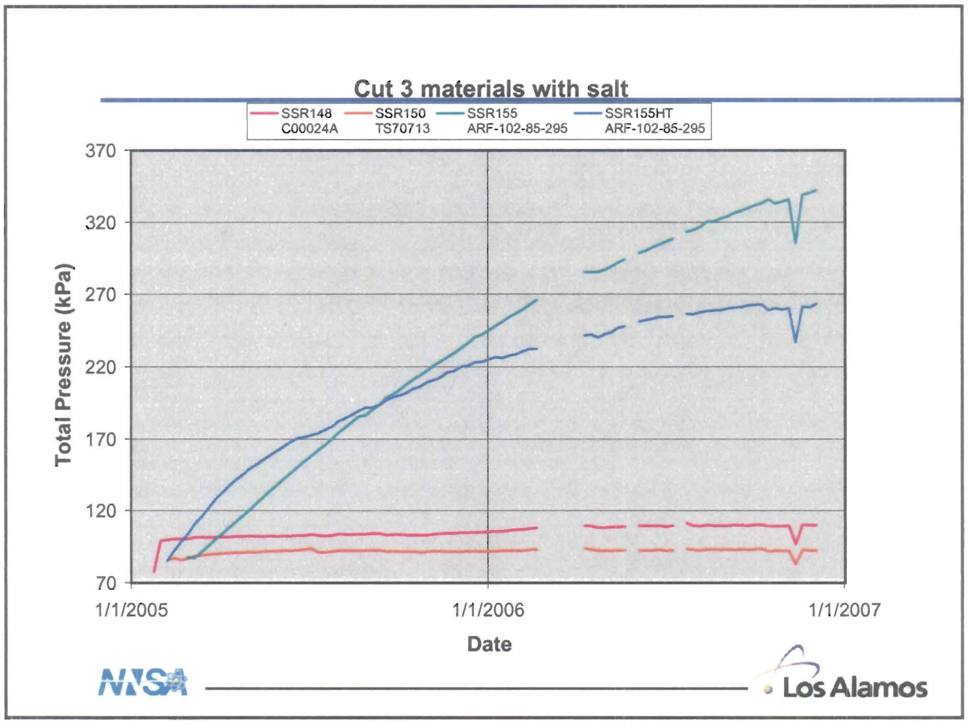
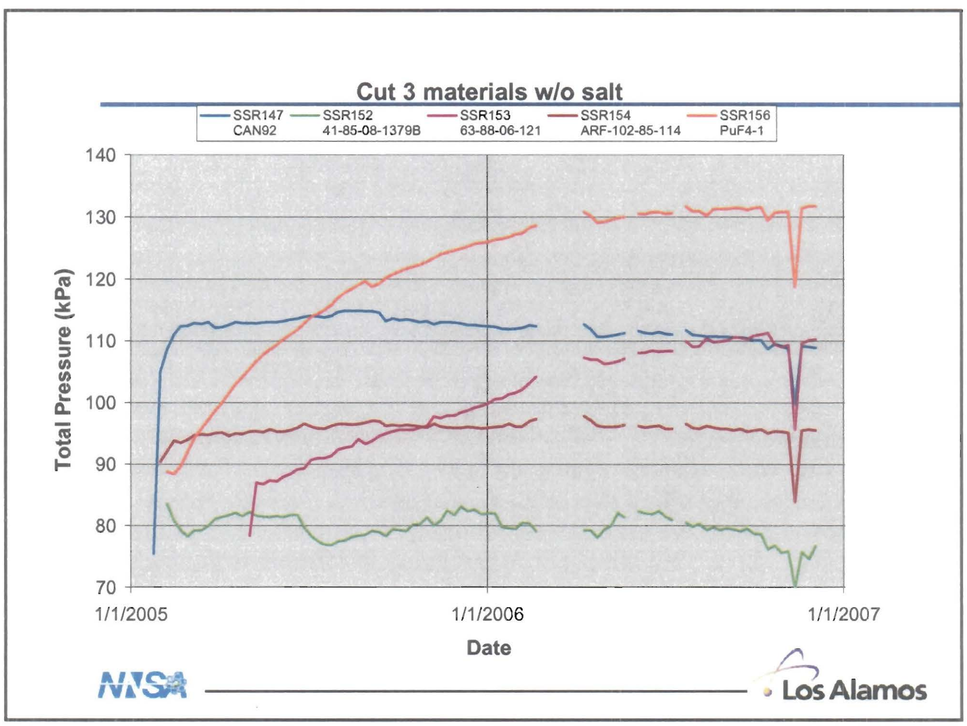
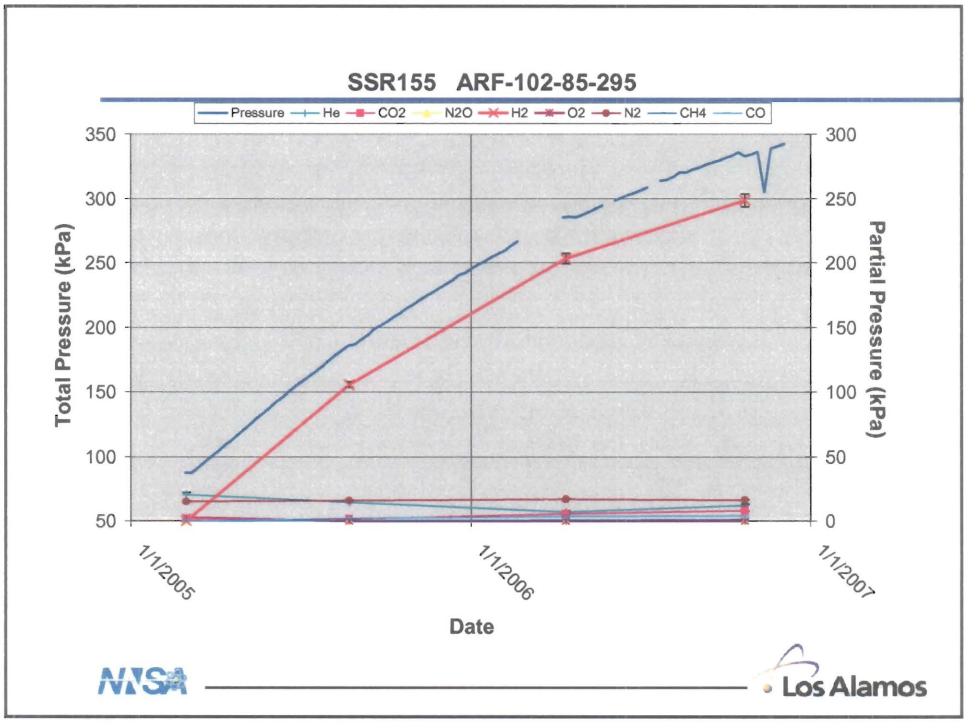

SSR137 11589
Pressure
He
CO2
N2O
H2
O2
N2
CH4
CO
Total Pressure (kPa)
Partial Pressure (kPa)
240
220
200
180
160
140
120
100
80
60
180
160
140
120
100
80
60
40
20
0
12/1/2003
11/30/2004
11/30/2005
11/30/2006
Date
NNSA
Los Alamos

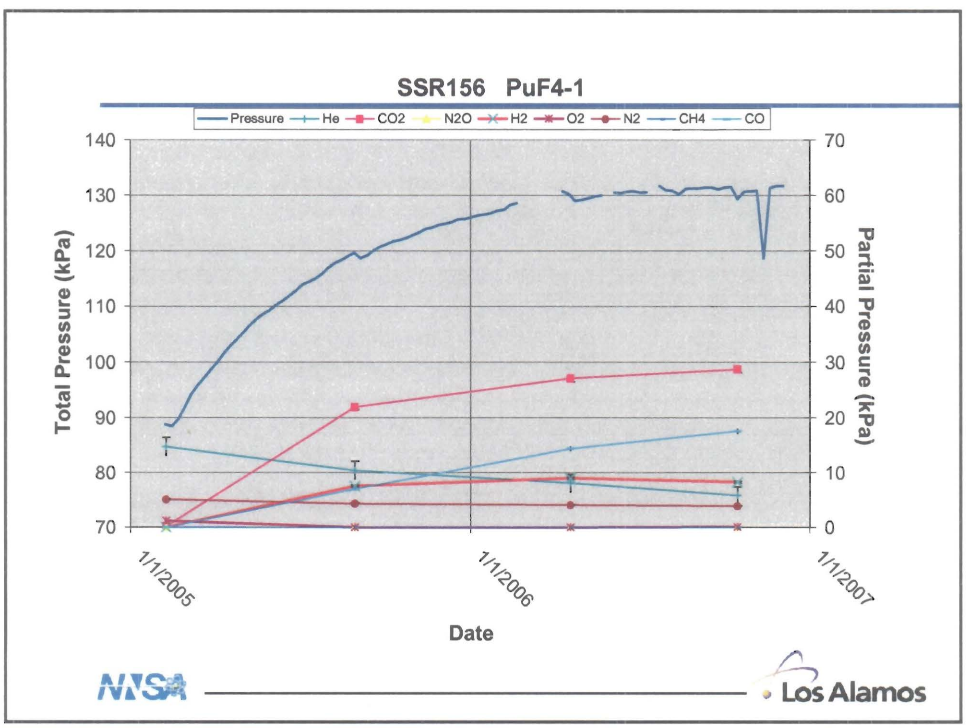

SSR143 ARF-102-85-355
Pressure
He
CO2
N2O
H2
O2
N2
CH4
CO
Total Pressure (kPa)
Partial Pressure (kPa)
100
95
90
85
80
75
25
20
15
10
5
0
12/1/2003
11/30/2004
11/30/2005
11/30/2006
Date
NNSA
Los Alamos


Cut 3 materials with salt
SSR148 C00024A
SSR150 TS70713
SSR155 ARF-102-85-295
SSR155HT ARF-102-85-295
Total Pressure (kPa)
370
320
270
220
170
120
70
1/1/2005
1/1/2006
1/1/2007
Date
NNSA
Los Alamos

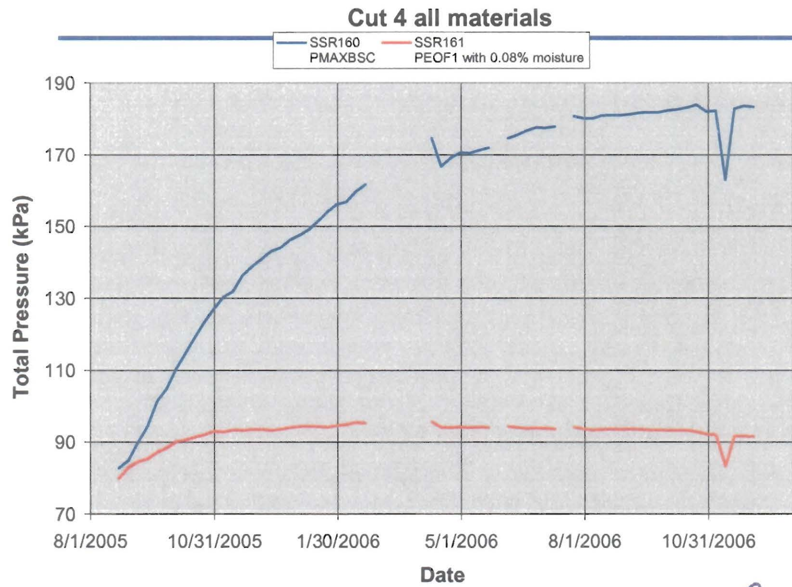

Cut 3 materials w/o salt
SSR147 CAN92
SSR152 41-85-08-1379B
SSR153 63-88-06-121
SSR154 ARF-102-85-114
SSR156 PuF4-1
Total Pressure (kPa)
140
130
120
110
100
90
80
70
1/1/2005
1/1/2006
1/1/2007
Date
NNSA
Los Alamos



SSR155 ARF-102-85-295
Pressure He CO2 N2O H2 O2 N2 CH4 CO
Total Pressure (kPa)
Partial Pressure (kPa)
350
300
250
200
150
100
50
300
250
200
150
100
50
0
1/1/2005
1/1/2006
1/1/2007
Date
NNSA
Los Alamos



SSR156 PuF4-1
Pressure He CO2 N2O H2 O2 N2 CH4 CO
Total Pressure (kPa)
Partial Pressure (kPa)
140
130
120
110
100
90
80
70
70
60
50
40
30
20
10
0
1/1/2005
1/1/2006
1/1/2007
Date
NNSA
Los Alamos



Calculation of H_2 gas generation rates

$$G = \frac{\Delta P}{\Delta t} \cdot CF$$

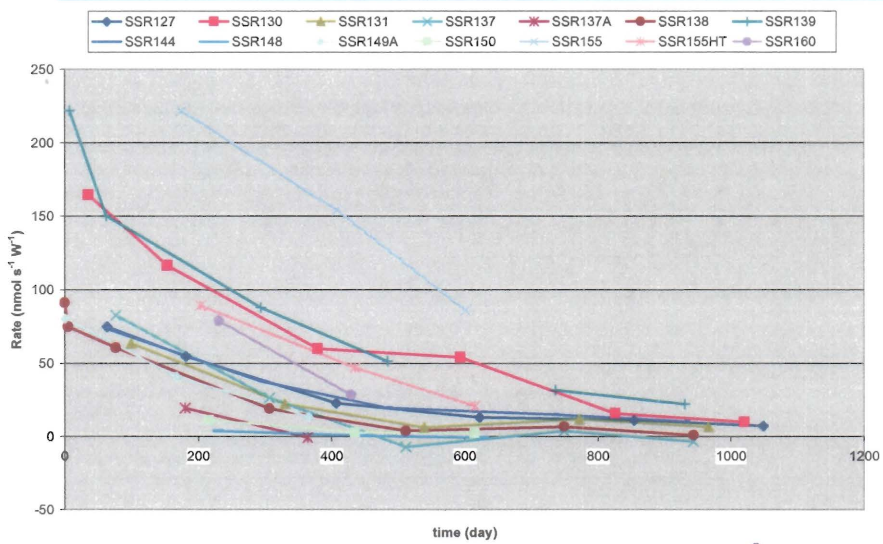
$$CF = \frac{Vgas}{RT} \cdot \frac{1}{86400\, seconds/day} \cdot \frac{1}{Dose(Watts)}$$

$$Vgas = 5ml - m/\rho$$

$$Dose = Wattage \cdot m \cdot f$$

where m is the mass of the sample, ρ is the sample pycnometer density, and f is the fraction of the total number of electrons that are associated with the water in the sample.

H_2 gas generation rates for salts



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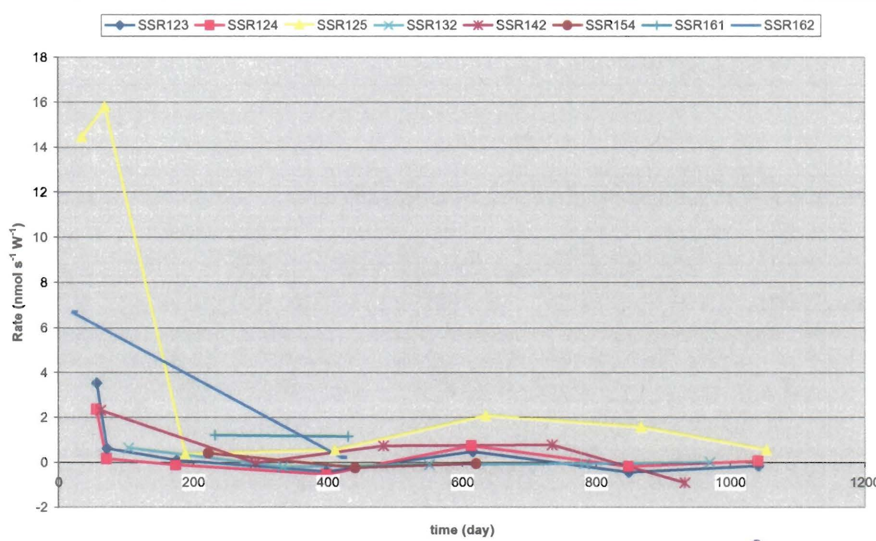
H_2 gas generation rates for pure oxides



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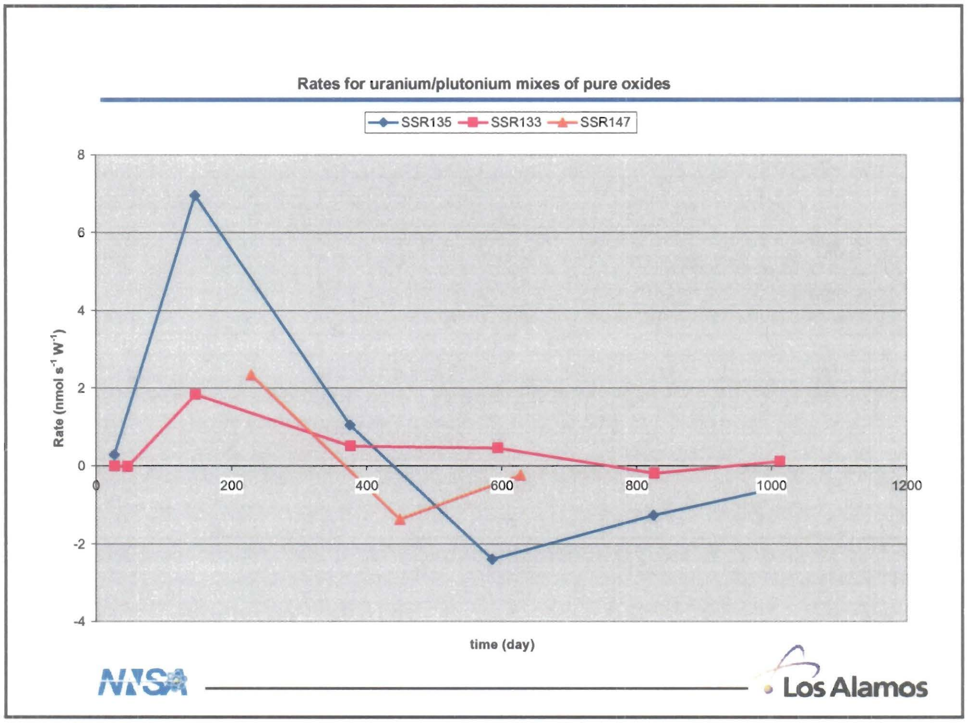

Rates for uranium/plutonium mixes of pure oxides
SSR135
SSR133
SSR147
Rate (nmol s^{-1} W^{-1})
8
6
4
2
0
-2
-4
0
200
400
600
800
1000
1200
time (day)
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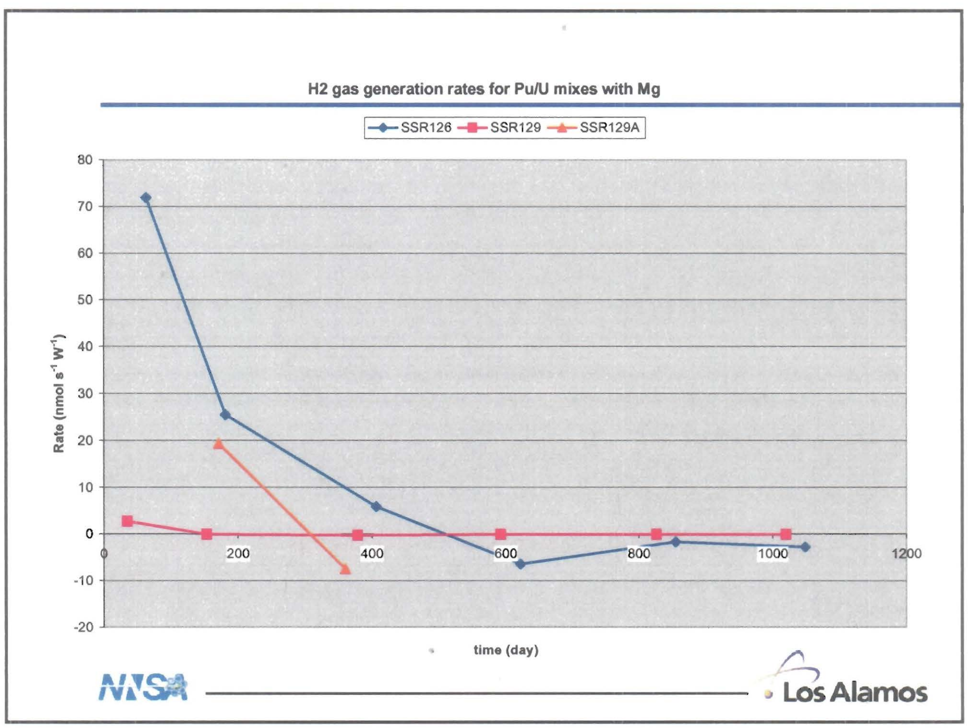

H2 gas generation rates for Pu/U mixes with Mg
SSR126
SSR129
SSR129A
Rate (nmol s^{-1} W^{-1})
80
70
60
50
40
30
20
10
0
-10
-20
0
200
400
600
800
1000
1200
time (day)
NNSA
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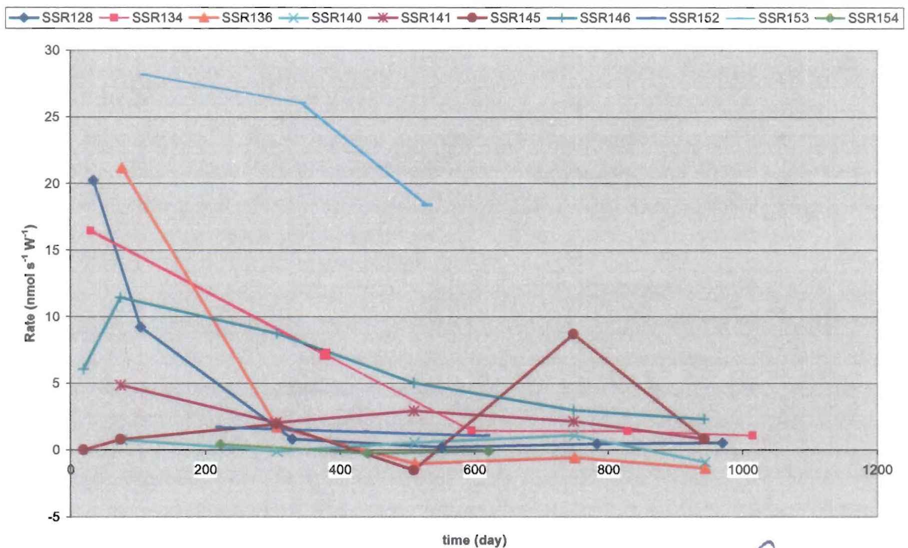
Hydrogen gas generatin rates for materials containing magnesium w/o chloride



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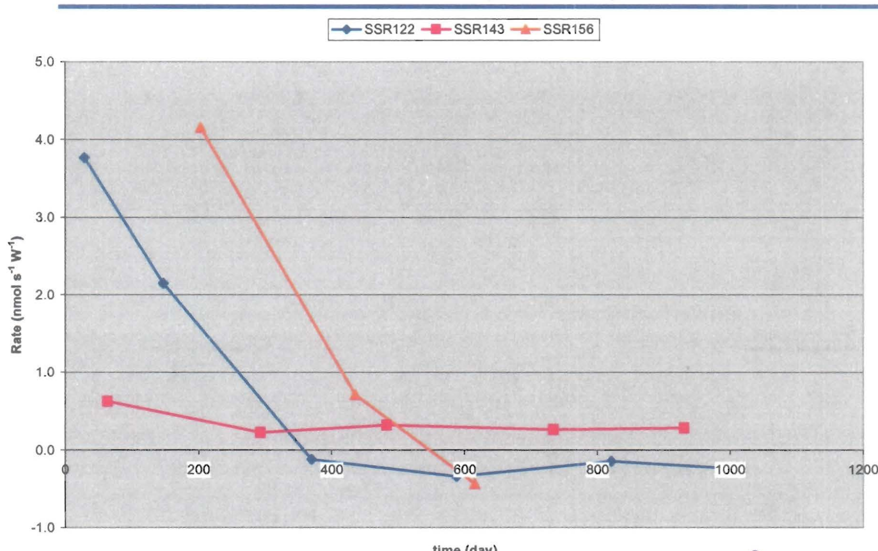
H2 gas generation rates for material containing Be and F as major contaminants



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Materials with ambiguous results

1. Materials that have been removed and could be reloaded.
   i. 7032282, a salt whose reactor leaked and was not reloaded.
   ii. C00695, a salt with an original high rate. Was reloaded but never attained original rate. Propose to start with new material if any exist.
2. Materials with leaking containers that could be reloaded.
   i. BLO-39-11-14-004, high wattage material, results ambiguous.
   ii. MISSTD1, high surface area material, reactor leaked.

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Materials that could be removed

Material	Number of containers	Retain	Remove
Salts	15	15	0
Oxides	8	3	5
U/Pu oxides	3	0	3
U/Pu with Mg	3	1	2
Be/F	3	2	1
Mg oxides	10	6	4
Total	42	27	15

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Current 45 position matrix status

Remove reactors with purple background

	A	B	C	D	E
1	11589 studies	139	129	153	11589 studies
2	144	146	131	129A	155HT
3	143	145	130	155	152
4	142	141	124	135	150
5	149A	140	123	133	154
6	137A	138	127	134	~~151~~156
7	160	137	126	125	162rep
8	161	136	122	132rep	148
9	blank	strip	strip	128	147

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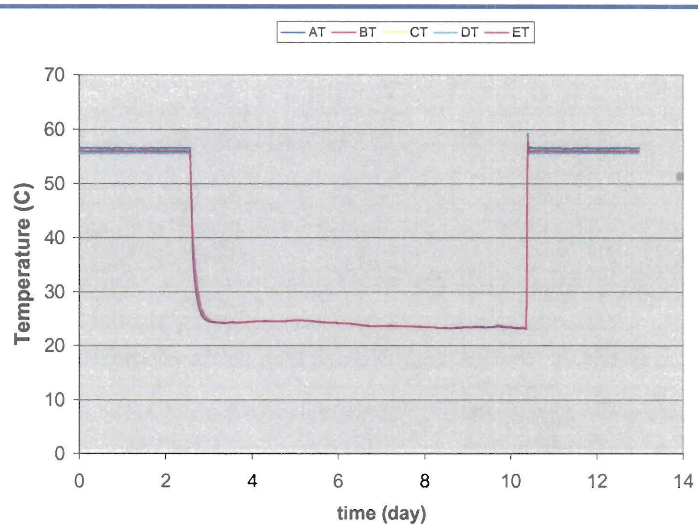
Temperature in small-scale array



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Water vapor pressure in containers

The loss of heating to the reactor matrix resulted in a reduction of pressure. Assume contributions from the ideal gas law and water vapor pressure.

$$\Delta P_{water} = \Delta P - P_1\left(1 - \frac{T_2}{T_1}\right)$$

$$RH = \frac{\Delta P_{water}}{\left(VP_{water,T_1} - VP_{water,T_2}\right)}$$

$$T_1 = (53 + 273)K$$

$$T_2 = (26 + 273)K$$

$$VP_{water,T_1} = 14.3kPa$$

$$VP_{water,T_2} = 3.4kPa$$

Estimated RH in all MIS small-scale reactors from $\Delta P/\Delta T$

SSR Number	MIS SAMPLE ITEM	RH	Material		SSR Number	MIS SAMPLE ITEM	RH	Material
SSR123	TS707001	84%	1 oxide		SSR153	63-88-06-121	44%	3 Mg w/o Cl
SSR124	5501579	76%	1 oxide		SSR141	7242141 (fur ball)	34%	3 Mg w/o Cl
SSR125	MT-1490	55%	1 oxide		SSR154	ARF-102-85-114-1	33%	3 Mg w/o Cl
SSR162	MISSTD	10%	1 oxide		SSR128	7242201	26%	3 Mg w/o Cl
SSR161	PEOF1	8%	1 oxide		SSR146	R438	15%	3 Mg w/o Cl
SSR142	PBO-47-09-012-023	4%	1 oxide		SSR140	7242165	15%	3 Mg w/o Cl
SSR132	BLO-39-11-14-004	4%	1 oxide		SSR145	64-85-12-1858	9%	3 Mg w/o Cl
SSR160	PMAXBS	41%	2 salt		SSR136	1000089	6%	3 Mg w/o Cl
SSR155	ARF-102-85-295	39%	2 salt		SSR152	41-85-08-1379B	-4%	3 Mg w/o Cl
SSR148	C00024A	37%	2 salt		SSR134	R441	-9%	3 Mg w/o Cl
SSR144	ARF-102-85-365	25%	2 salt		SSR143	ARF-102-85-355	57%	4 Be/F
SSR155HT	ARF-102-85-295	21%	2 salt		SSR156	PuF4-1	15%	4 Be/F
SSR150	TS707013	17%	2 salt		SSR122	7161856	7%	4 Be/F
SSR137	11589	15%	2 salt		SSR129A	5501407	32%	5 U/Pu Mg
SSR131	ARF-102-85-223	11%	2 salt		SSR129	5501407	17%	5 U/Pu Mg
SSR138	53038	6%	2 salt		SSR126	669194	15%	5 U/Pu Mg
SSR127	CLLANL025	4%	2 salt		SSR135	SCP711-56	19%	6 U/Pu oxide
SSR130	C06032A	2%	2 salt		SSR147	CAN92	1%	6 U/Pu oxide
SSR151	7032282	0%	2 salt		SSR133	PSU-84-06-05	0%	6 U/Pu oxide
SSR149A	C00695	-8%	2 salt					
SSR139	520610020	-15%	2 salt					
SSR137A	11589	-18%	2 salt					

Discussion of estimated RH

- Expect pure oxides to have high RH. Highest RHs are oxides, however a range is observed.
- Expect salts to have low RH. Lowest RHs are salts however, a range is observed.
- Uranium containing materials may have trend to lower RH.
- If corrosion occurs at higher RHs, then the containers with the lower RHs may have minimal corrosion.
- The lack of any consistency across materials may indicate that the approach lacks merit. A measurement at unloading may be useful.

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Mathematical modeling of the H_2 gas generation over time

A method to more realistically calculate maximum pressures for 3013 inner containers based on information in the MIS Database is desired. Monitoring of the small-scale reactors over many years have now shown that the linear extrapolation of the initial rates observed in the small-scale reactors is too conservative. Known physical processes such as depletion of the available water and hydrogen gas reactions with materials are not taken into account in the linear extrapolation. A simple model has been developed that incorporates these processes and is demonstrated using some of the data.

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Approach to mathematical modeling of the H_2 generation

$H_2O \rightarrow H_2$

the G-value of H_2 gas generation is constant which means the H_2 gas generation rate due to radiolysis of water decreases as the water content declines. Assuming one water is lost for each H_2 produced, one can calculate the reduction in H_2 gas generation rate. Defining

$C = G \cdot SA \cdot f\_E \cdot MW_{H2O}$

Where G is the G-value for H_2, SA is the specific activity in W/kg, f_E is the fraction of the number of electrons in the material due to water, and MW_{H2O} is the molecular weight of water.

$n_{H2O} = n_{H2O,initial} \exp(-Ct)$ and $n_{H2} = n_{H2O,initial}(1-\exp(-Ct))$

where n_{H2O} is the moles of water in the material at time t and $n_{H2O,initial}$ is the starting number of moles of water. n_{H2} is the moles of H_2 at time t from radiolysis.

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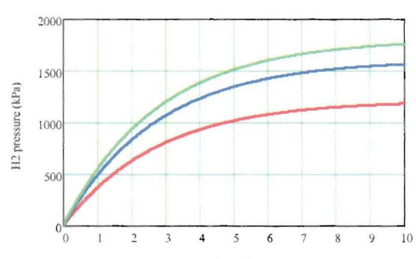
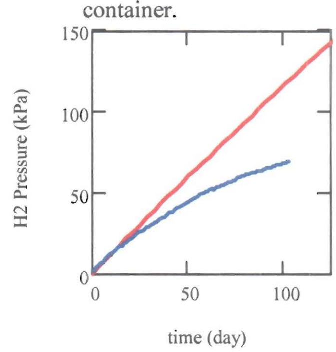
Example: use data from Large-Scale corrosion cans.

Hydrogen pressure vs time accounting for loss of water in the three corrosion containers. G = 260 nmol/(s W).



Comparison of the model and data for the first corrosion container.



H_2 gas generation due to loss of water does not turn over nearly as fast as the actual hydrogen pressure. One possible fix is a loss term for hydrogen.

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Adding a loss term for H_2 gas.

$H_2O \rightarrow H_2$

H2→OH or some other sink on the surface.

Does not produce water because there is no O_2 in gas phase.

$dn_{H2}/dt = C\, n_{H2O} - k\, P_{H2}$

$$P1\_H2\_model(t) := \frac{C}{k \cdot \frac{R\_gas \cdot T}{Vgas} - C} \cdot \frac{R\_gas \cdot T}{Vgas} \cdot n1\_H2O \cdot \left(\exp(-C \cdot t) - \exp\left(\frac{-k \cdot R\_gas \cdot T}{Vgas} \cdot t \right) \right)$$

where k is a constant to be determined and may be material dependent. For the MasterBlend material k is determined by a fit to the data to be

$$k := 7.5 \cdot \frac{mol}{s} \cdot \frac{10^{-11}}{kPa}$$

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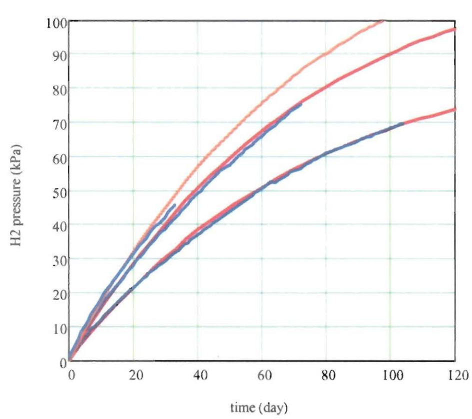
Results for the three Large-Scale corrosion containers

Data – blue Model - red



Same G of 330 nmol/s W and same rate of H_2 loss (7.5×10^{-11} mol/s kPa). G is similar to the maximum G seen in the small scale experiments. The differences between the curves are due to the water content.

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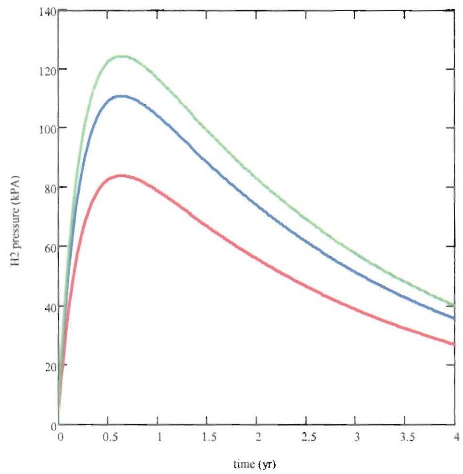
Long time behavior of the hydrogen pressure



The maximum hydrogen pressure from the model is now less than 130 kPa. At long times, as the rate of H_2 loss exceeds the rate of H_2 production, the H_2 pressure decreases.

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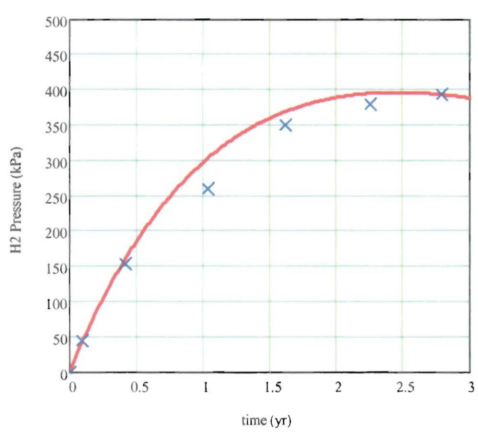
Example: SSR130

Data – blue   Model - red



G – 164 nmol/s W

k – 2.8x10^{-14} mol/s kPa

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Conclusions

A simple model for H_2 gas generation has been developed. It includes

i. H_2 gas generation based on fundamental G-values
ii. accounts for loss of water from radiolysis
iii. a loss term for H_2

The model reproduces the shape of the hydrogen gas generation curve with time. For multiple samples of the same material, it correctly incorporates the dependence upon water. By adopting appropriate maximum G values and minimum loss terms the model would improve upon existing estimates of the H_2 pressure with time.

Work to be done.

Need to analyze all of the existing data using the model.

Develop conservative estimates of the G value and k for classes of materials.

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Microscopic Study of the Deliquescence Behavior of Binary and Ternary Salts

Ed Garcia[†], Obie Gillespie[†], Steve Joyce[‡], Kirk Veirs[†], Laura Worl[†]
[†]Plutonium Manufacturing Technology Division, [‡]Chemistry Division
Los Alamos National Laboratory

Four salts, $KMgCl_3$, K_2MgCl_4, $K_3NaMgCl_6$, and an ER simulant, were examined in an Environmental Scanning Electron Microscope (ESEM) capable of imaging at relative humidities ranging from ~0% up to 95%. Spatially-resolved x-ray fluorescence was employed to determine sample stoichiometry, impurities and phase separation.

Salts were prepared from reagent grade NaCl, KCl, and anhydrous $MgCl_2$ in a He inert glove box by calcination to 850 °C for one hour. Powder x-ray diffraction verified the final product (to 5%).

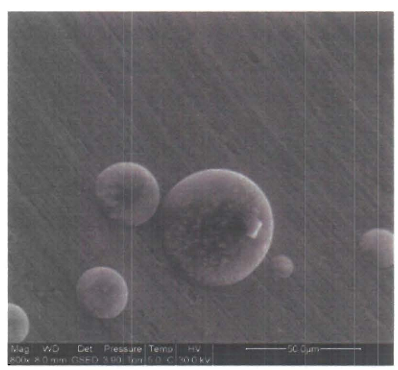
$KMgCl_2$: Carnallite

Most particles have correct stoichiometry. Occasional MgO impurities.

Initial deliquescences occurs at ~57%RH. Note crystallites in droplet.

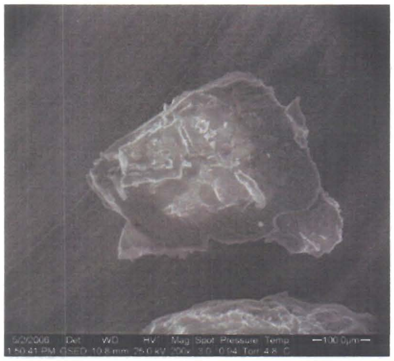
K_2MgCl_4

All particles have correct stoichiometry. No impurities.

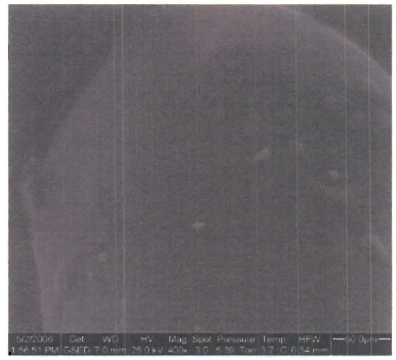


Deliquescences occurs at ~57%RH. Note crystallites in droplet.

$K_3NaMgCl_6$

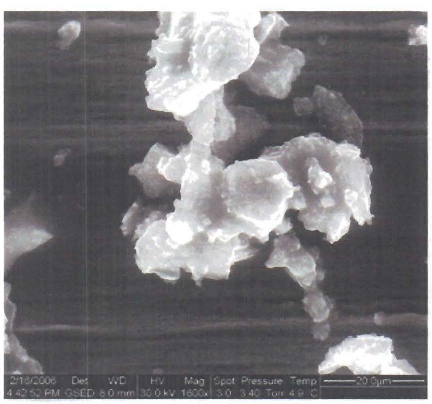


Most particles have correct stoichiometry. Occasional Al and Si impurities.

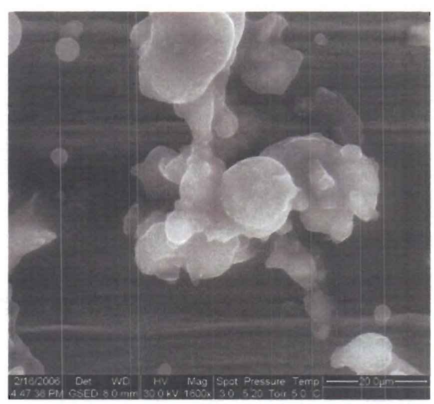


Initial deliquescences occurs at ~57%RH.

Na/K Cl (95%) $MgCl_2$ (5%) “ER Salt”



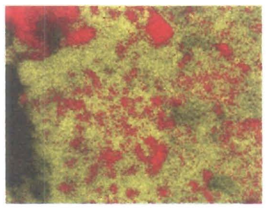
Sample is chemically heterogeneous.



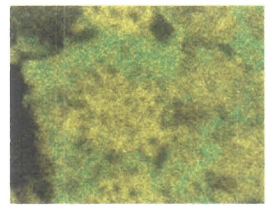
Initial deliquescences occurs at ~57%RH. Note crystallites in droplet.

"ER Salt" Clearly Inhomogeneous Prior to Deliquescence

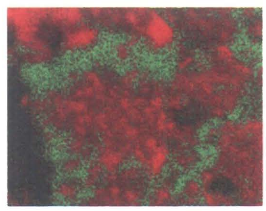
"Average" Composition: K 22%, Na 28%, Mg 3%, Cl 47%



K and Na

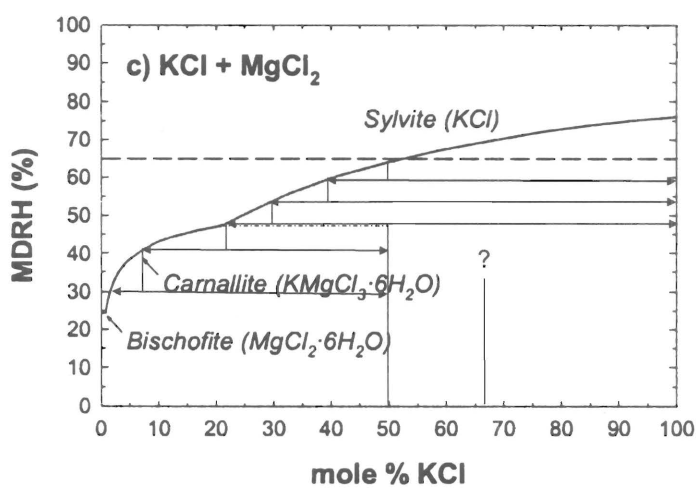


K and Mg



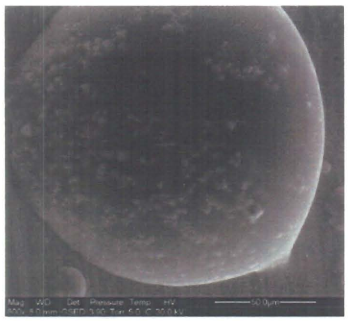
Mg and Na

Precipitation of a deliquesced Carnallite yields Sylvite, Carnallite, and Bischofite †

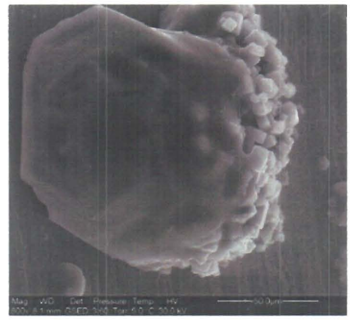


† Diagram from Pabalan, Yang, and Browning, Mat. Res. Soc. Symp. Proc. Vol. 713, pg. JJ1.4.4, (2002).

Phase Separation of Binary Salt after Drying from Initial Deliquescence (K_2MgCl_4)



Salt just above DRH



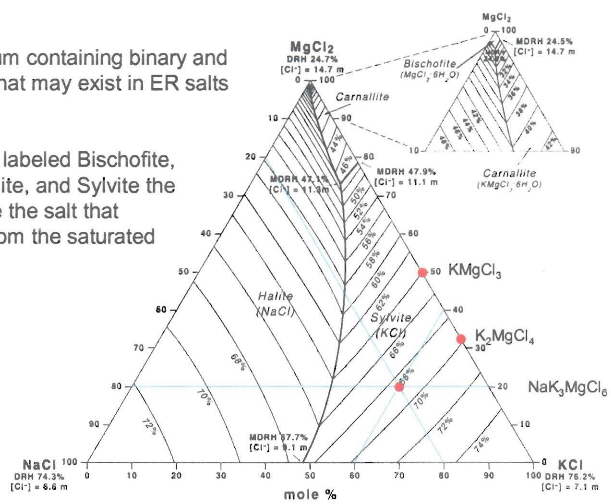
Salt just after crystallization from drop. Cubes on right are KCl, Carnallite crystals on left.

Phase separation to Carnallite, KCl and $MgCl_2$

Ternary phase diagram constructed from saturated solution experimental data†

The magnesium containing binary and ternary salts that may exist in ER salts are indicated.

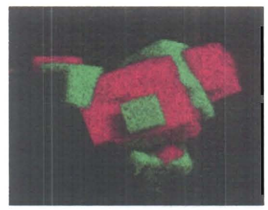
In the regions labeled Bischofite, Carnallite, Halite, and Sylvite the labels indicate the salt that precipitates from the saturated solution.



† Diagram from Pabalan, Yang, and Browning, Mat. Res. Soc. Symp. Proc. Vol. 713, pg. JJ1.4.4, (2002).

Phase Separation of "ER Salt" After Complete Hydration (~80%RH) And subsequent Dehydration

Secondary Electron Image



K and Na Map

Phase separation to KCl, NaCl and $MgCl_2$

Increase of water content upon deliquescence by these hydrated salts from solubility data[†].

Compound		Saturated solution
$MgCl_2 \cdot 6H_2O$	→	9 waters per Mg
$KMgCl_3 \cdot 6H_2O$	→	28 waters per Mg
$K_2MgCl_4 \cdot 6H_2O$	→	37 waters per Mg
$NaK_3MgCl_6 \cdot 6H_2O$	→	60 waters per Mg

[†]R. T. Pabalan and K. S. Pitzer, Geochimica et Cosmochimica Acta ***51***, 2429-2443 (1987).

Conclusions

1. The magnesium component of ER-like salts is NaK_3MgCl_6 (from earlier work by E. Garcia).
2. The magnesium component most likely forms a hexahydrate upon exposure to water vapor (from the pattern for $MgCl_2$ and $KMgCl_3$).
3. The binary and ternary salts deliquesce above 57% RH.
4. The binary salts phase separate upon precipitation.
5. The amount of water absorbed by the material upon deliquescence (normalized to the magnesium content) increases in the series $MgCl_2$, $KMgCl_3$, $K2MgCl_4$, NaK_3MgCl_6.
6. Report currently in draft form.
7. Calcium chloride may also form binary and ternary salts whose water uptake and deliquescence behavior may be important.

Application of Pressure Calculations to the ISP Database

Larry Peppers

Gary Friday

January 2007

Why Have Estimated Container Pressures

- Objective —
  - Estimate total pressure in 3013 containers for surveillance
  - Estimate gas composition
    - Hydrogen
    - Oxygen
    - Others – CO_2, N_2, … etc.
  - Establish a relative ranking of containers based on pressure

MIS Items Pressure Behavior

- Pressure behavior can be categorized into four groups
  - Items with chloride, with or without magnesium
    - Primarily generate hydrogen
    - Highest pressures
    - Hydrogen model applies
  - Items with high magnesium, no chloride
    - Small amounts of hydrogen
    - Other gasses at concentrations on the same order of magnitude as hydrogen
    - Generally low pressures (less than 150kPa)
    - Hydrogen model applies only to part of the total pressure
  - Pure oxides
    - Very little gas generation
    - Pressures around 90-160kPa
    - Very little hydrogen
  - Impure oxides that act like pure oxides
    - Impurities do not influence gas generation

Past Attempt at Calculating Container Pressure

- Pressure calculation based on initially observed gas generation rates (G-values)
- G-values assigned to four groups
  - Items with chloride = 1.1
  - Items with high magnesium = 0.4
  - Items with uranium = 1.2
  - Pure oxides = 0.1
- Over estimated pressures

$$\left[\frac{\Delta n}{\Delta t}\right] = G\, Q_{H_2O}\, 103.6$$

$$Q_{H_2O} = Q\,\frac{\# e^-_{H_2O}}{\# e^-_{mat}}$$

$$\left[\frac{\Delta P}{\Delta t}\right] = \frac{\left[\frac{\Delta n}{\Delta t}\right] R\, T}{V_{gas}}\; 8.64\times10^{-5}$$

Pressure Model for Hydrogen

Pressure as a function of time (items with chloride):

$$P_{H_2}(t) = \frac{C}{k \cdot \frac{R\,T}{V_{gas}} - C} \cdot \frac{R\,T}{V_{gas}} \cdot n_{H_2O} \left(e^{(-C \cdot t)} - e^{\left(-k \cdot \frac{R\,T}{V_{gas}} \cdot t\right)} \right)$$

Where:

$$C = G \cdot SA \cdot f_{e-H_2O} \cdot MW_{H_2O}$$

$$k = 7.5\times10^{-11} \frac{mol}{s \cdot kPa}$$

$$MW_{H_2O} = 0.018 \frac{kg}{mol}$$

$$G = 200 \frac{nmol}{W \cdot s}$$

$$SA = \text{Specific Activity} \frac{W}{kg}$$

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Pressure Model for Hydrogen

Where:

$$f_{e-H_2O} = \frac{\frac{10}{18}}{f_{PuO_2}\frac{110}{271} + f_{U_3O_8}\frac{340}{833} + f_{NpO_2}\frac{109}{269} + f_{AmO_2}\frac{111}{275} + f_{salts}\frac{1}{2} + f_{H_2O}\frac{10}{18}}$$

$$T = 3\,Q + 1.6\,Q + 25 + 273$$

Q = Container Wattage

$$n_{H_2O} = \frac{m \cdot f_{H_2O}}{MW_{H_2O}}$$

m = Net Weight (kg)

f_{H_2O} = fraction water

$$V_{gas} = V_{container} - \frac{m}{\rho} - \frac{m_{CC}}{\rho_{SS}}$$

$$\rho = \frac{1}{\frac{f_{PuO_2}}{\rho_{PuO_2}} + \frac{f_{U_3O_8}}{\rho_{U_3O8}} + \frac{f_{NpO_2}}{\rho_{NpO_2}} + \frac{f_{AmO_2}}{\rho_{AmO_2}} + \frac{f_{H_2Od}}{\rho_{H_2O}} + \frac{f_{salt}}{\rho_{salt}}}$$

Example Calculation

- Pu% = 70.643
- Am% = 0.109
- H_2O% = 0.2887
- m = 3333.7g
- m_{cc} = 1635.0 g
- Q = 5.9468 watts
- $V_{container}$ = 2.068 liters
- Weld date = 7/2/2003
- t = 580 days

- FY 05 Pressure
  - 255 kPa
- FY 07 Pressure
  - 42 kPa

Application of the model to the database

- Three ways to apply the model to the ISP Database
  - Through the surveillance bin assignment
    - Pressure and corrosion bin assumed or known to contain chlorides
    - Pressure and Innocuous bin not calculated
  - Through the MIS Represented group
    - Not chloride containing items would be included
  - Through Prompt gamma analysis results
    - Not all items have prompt gamma data
- Limitations
- Accuracy of assigning 3013 containers to the correct model

Path Forward

- Refine model for items with high magnesium without chloride
  - G and k values specific to high magnesium
- Define pressures for other MIS groups
- Define scheme to be used in the database
- Implement equations and application scheme into the ISP database
  - End of February 2007
- Reassess model when DE data is available for 3013 containers

Conclusions

- Hydrogen model can calculate H_2 in salts with reasonable results
- The model overestimates non-chloride materials
- Limited to the accuracy of the measured moisture
- Implementation scheme in the ISP database needs further definition
- End of February 2007
- Reassess model when DE data is are available for 3013 containers

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Linkage of MIS / ISP Databases using MIS Representation

Larry Peppers

Josh Narlesky

January 2007

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Scope

- Working session to define methods and path forward
- Approval process
- Verify that MIS items are accurately assigned to process groups
- Modify the master represented table based on current information
- Verify that 3013 containers are accurately assigned to represented groups

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Background

- Each site submitted a representation document as part of 20 points requirements
- MIS items assigned to represent material from each process
- Four represented documents combined and used to connect the ISP database (3013 container) to the MIS items database (material representing container)
- Case studies indicate changes are necessary to the represented table to accurately connect the ISP database to the MIS items database

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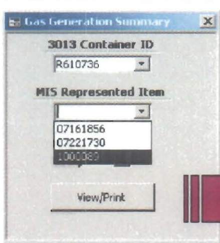
MIS Items

- Represent 3013 containers by process of origin
- Contain detailed characterization data for each process group
- Most processes are represented by at least one MIS item
- Prompt gamma represents some material
  - MIS Items Prompt gamma signatures grouped
  - 3013 containers Prompt gamma signatures matched to prompt gamma group

3013 Containers

- Process of origin defined for most original input items to the 3013 stabilization process
- Limited characterization data available
  - Most oxides have prompt gamma analysis
  - Some items have chemistry analysis
  - Some history information is suspect or missing
    - Assigned to the wrong IDC (RFETS)
- 3013 containers assigned to one or more representation groups
  - RFETS material mixed across representation groups to meet Material Disposition
  - Assignment process subject to accuracy of characterization information
- 3013 containers connected to MIS items through the representation table

Final Results in ISP Database



MIS Small-Scale Surveillance Summary

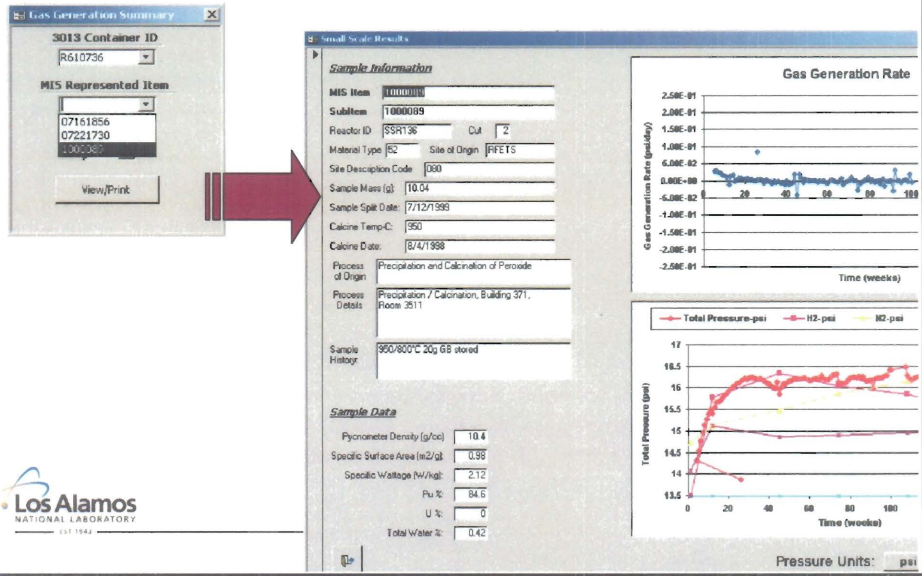
3013 Container ID: R610736

Represented by MIS Item: 1000089

Reactor Number SSR136

Week	Date	Total Pressure (psi)	CO2	N2O	He	H2	O2	
			Partial Pressure (psi)					
1	3/24/2004		1.55E-03 ± 5E-04	1.50E-03 ± 5E-04	1.11E+01 ± 2E-01	1.94E-04 ± 6E-05	4.93E-01 ± 1E-02	1.0
12	6/8/2004	16.4	1.69E-03 ± 6E-04	2.01E-01 ± 7E-03	1.07E+01 ± 2E-01	1.95E+00 ± 4E-02	1.41E+00 ± 3E-02	1.41
45	1/24/2005	15.8	1.76E-03 ± 6E-04	2.10E-01 ± 8E-03	1.08E+01 ± 2E-01	2.43E+00 ± 5E-02	1.18E+00 ± 3E-02	1.6
74	8/17/2005	16.1	1.80E-03 ± 6E-04	2.01E-01 ± 7E-03	1.09E+01 ± 2E-01	2.17E+00 ± 4E-02	1.21E+00 ± 3E-02	2.0
108	4/12/2006	16.3	1.71E-03 ± 6E-04	2.03E-01 ± 7E-03	1.09E+01 ± 2E-01	2.01E+00 ± 4E-02	1.25E+00 ± 3E-02	2.27
138	10/24/2006	16.0	1.89E-03 ± 6E-04	1.91E-01 ± 7E-03	1.10E+01 ± 2E-01	1.69E+00 ± 3E-02	1.26E+00 ± 3E-02	2.4

Final Results in ISP Database



Approval Process

- Changes need made to the represented table
- Who needs to approve changes to the master represented table?

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Case studies

- Items similar to 011589A
- Items similar to C06032A
- Assessment of RFETS group 1A (for 3013 container R601957)

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Specific Examples

- 011589A
  - 011589A should be added to Hanford group 1E for material with Prompt gamma peak ratio's like foundry oxide
  - Divide Hanford group 1A into pyrochemical and foundry oxide based on PG peak ratio
  - Remove 011589A from SRS group 1E and 2A
  - RFETS group 1B could be split into two groups based on date
    - 010xxx, 011xxx type material
    - TS707xxx material
- C06032A
- RFETS group 1A

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An Overview of 3013 Binning and Sample Selection and *The Path Forward*

Jerry Stakebake

Larry Peppers

Los Alamos National Laboratory

January 2007

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Initial Binning & Sample Selection

- Identified three categories for binning 3013 items based on potential failure mechanisms
- Developed a decision tree for binning
- Initial 3013's were binned using the decision tree and engineering review
- Developed a statistical protocol for sample selection
  - Pressure and Innocuous sampling 5-year schedule
  - Pressure and Corrosion sampling 10-year schedule
  - NDE containers greater than 3 years old
  - DE containers greater than 5 years old

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Container Selection for 2005 NDE

- Hanford – 23 + 10 additional supplemental
  - Pressure & Corrosion – 5 random
  - Pressure – 12 random
  - Innocuous – 1
  - Judgmental – 5
- SRS – 27
  - Pressure & Corrosion – 5 random
  - Pressure – 12 random
  - Innocuous – 1
  - Judgmental – 9
- LLNL – 2
  - Pressure & Corrosion – 1 random
  - Pressure – 1 random

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What has Changed since 2005?

- SRS packaging completed
- Reanalysis of RF TGA/FTIR moisture data and use of the FTIR data for binning decisions
- Additional PG and analytical data available
- Revision of PG concentration data for F to match the PG detectable Cl concentration
- Hanford weight gain data included in the data base

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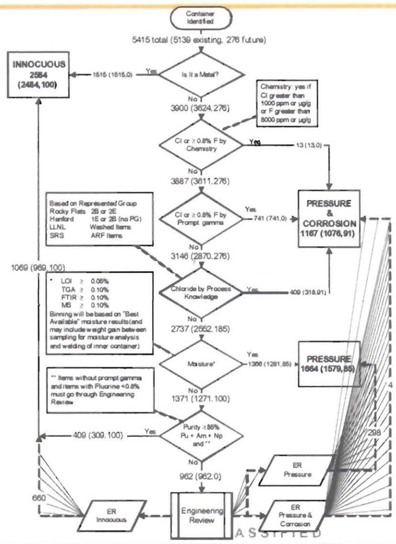
2006 Binning Results for all Containers



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2006 Binning Results for all Containers
[*Changes between 2005 and 2006]

Site	Innocuous	Pressure	Pressure And Corrosion	Total
Rocky Flats Packaged	 808 (+22)*	 721 (+115)	 359 (-144)	 1888 (-7)†
Hanford Packaged Unpackaged	 921 (-23) 0 (-9)	 746 (+160) 0 (-9)	 590 (-137) 0	 2257 0 (-18)
LLNL Packaged Unpackaged	 9 (+2) 0	 9 (+4) 0	 56 (-6) 61 (0)	74 (0) 61 (0)
SRS Packaged Unpackaged	 746 (-3) 0 (-41)	 103 (+103) 0 (-150)	 71 (+71) 0 (-100)	 920 0 (-120)
LANL Packaged Unpackaged	 12 88 (-68)	 18 67 (+60)	 2 28 (-119)	 32 183 (-127)
Total	2584 (-120)	1664 (+283)	1167 (-435)	5415 (-272)

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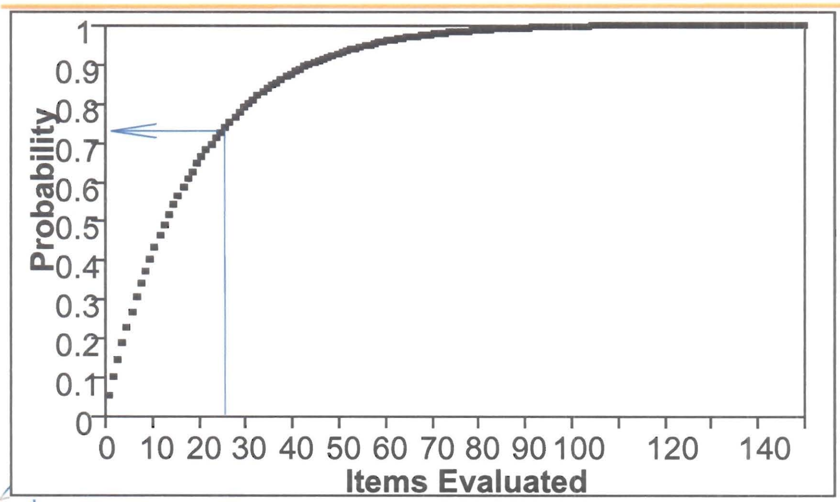
Surveillance Sample Selection

- Statistical random samples needed to meet 99.9%/5% based on bin population

Site	Pressure and Corrosion	Pressure
Hanford	66	61
LLNL	13	1
Rocky Flats	40	59
SRS	8	8
LANL	4	1
TOTAL	131	130

Probability of detecting at least one item from the worst 5%.



Other Sample Selection Protocol

- Judgmental sample selection from Pressure and Corrosion and Pressure bins
  - These samples augment statistical samples
  - Selection made with engineering judgment based on
    - Shelf-life studies
    - Comparison of statistical sample to the population
    - Field surveillance results
    - Targets containers with greatest potential for degradation
- Sample selection from Innocuous Bin
  - Ten total samples to come from three strata of innocuous sample binning
    - Oxides with NO ER
    - Oxides with NO ER and NO F
    - Oxides with both ER and F

Container Selection for 2006 NDE

- Rationale for sample selection
  - Samples identified using 2005 binning results
- 2006 [all sites]
  - Pressure Bin – 25
  - Pressure and Corrosion Bin – 11
  - Innocuous Bin - 2
  - Judgmental Samples – 9
- Additional samples of opportunity for 2006
  - Hanford – 27
  - Rocky - 2

Container Selection for NDE

- 2007 [all sites]
  - Pressure Bin – 25
  - Pressure and Corrosion Bin – 2
  - Innocuous Bin – 2
  - Judgmental Samples – 4
- 2008-2009 [all sites]
  - Pressure Bin – 25 per year

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Container Selection for DE

- Pressure and Corrosion Bin
  - 2007
    - Random – 3
    - Judgmental – 4
  - 2008-2016
    - Random – 13 per year
  - 2017
    - Random – 11
- Pressure Bin
  - 2007
    - Random – 2

Future Directions and Requirements

- Completion of packaging at LANL and LLNL may result in adjustments to binning and statistics
- Additional PG measurements/reanalysis and reanalysis of TGA moisture may change bin assignments
- Selection of 2 containers in Pressure bin for DE will be selected per year in 2008-2009 based on engineering judgment
- Additional judgmental samples may be selected for DE or NDE based on field surveillance and shelf studies
- Feedback loop from DE sample analysis
- Validation of moisture assumption on binning Innocuous Items

Comprehensive Nuclear Material Packaging and Storage Plan

January 25,2007
SRS

Approach

- Risk rank containers
- Repackage and/or disposition of non-standard packages. Include all items not already in 00-1.
- Surveil and monitor pristine packages in TA-55
- ISD Team address problem items, reprioritize, collect data from repackaging to validate and refine risk ranking
- Control all aspects of container use.

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Scope

Material Location Site (non-glovebox)	All Containers	Non-Standard Containers	% of Total Remaining Risk
TA-55 Basement	6491	2204	75
TA-55 Main Floor	1033	701	24
TA-55 Yard	14	14	< 1
TA-55 SST Pad	29	27	< 1
TA-18	451	399	<1
TOTAL	8018	3345	100

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Slide 3

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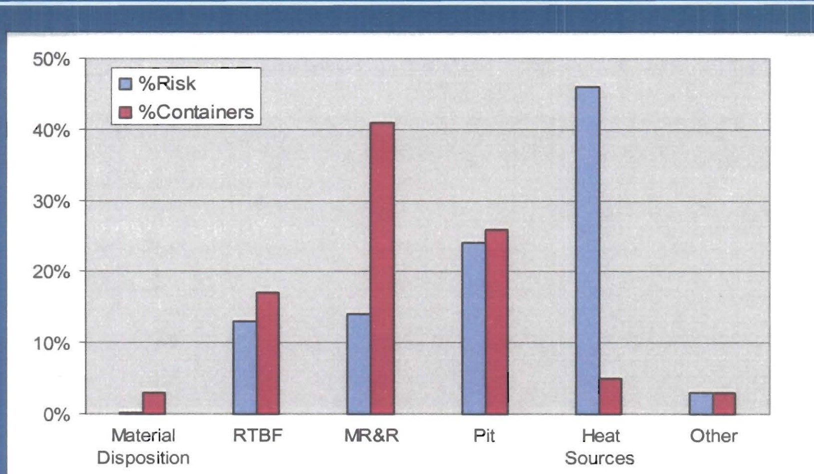
Risk & Containers by Program



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Compensatory Measures from Vault Incident

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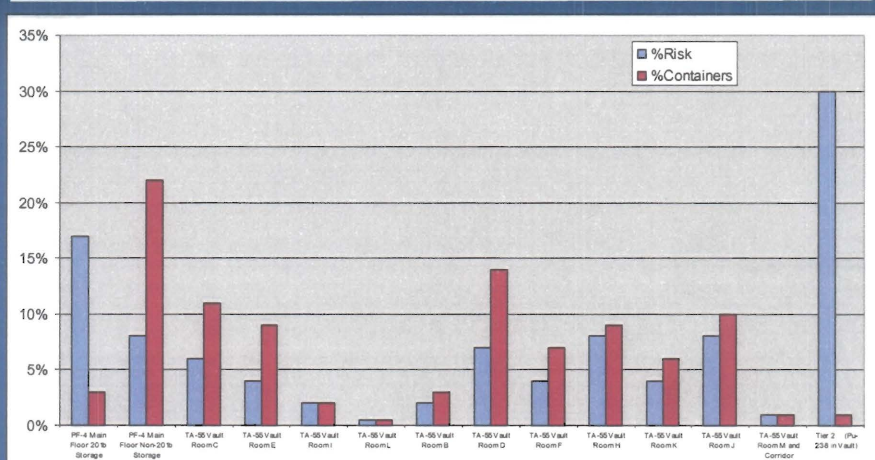
Risk & Containers by Location



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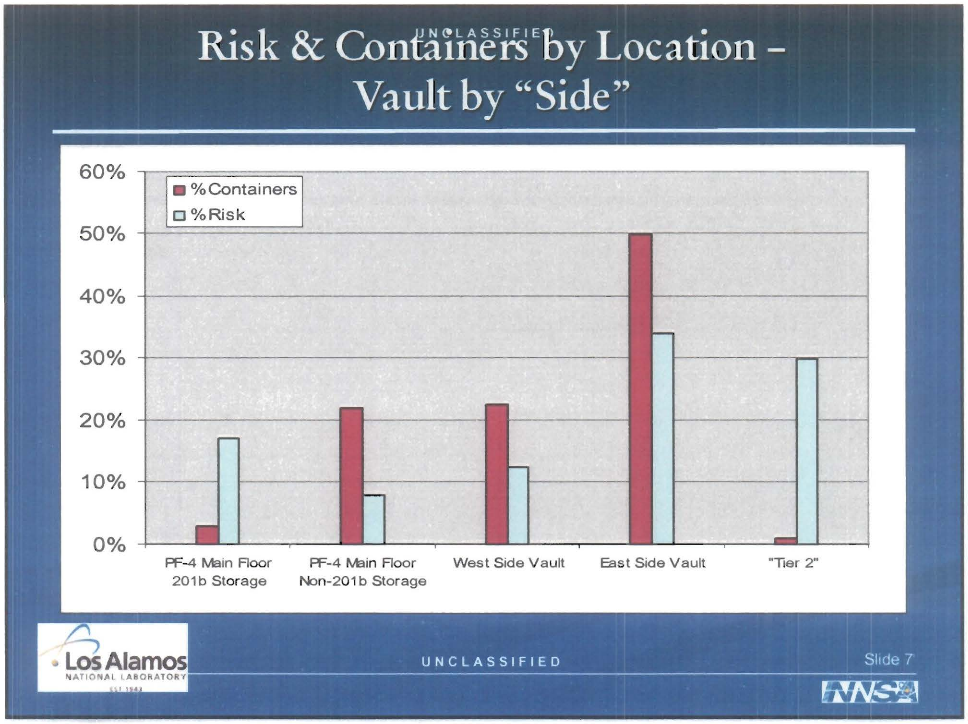
Slide 6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Risk & Containers by Location –
Vault by "Side"
60%
50%
40%
30%
20%
10%
0%
%Containers
%Risk
PF-4 Main Floor
201b Storage
PF-4 Main Floor
Non-201b Storage
West Side Vault
East Side Vault
"Tier 2"
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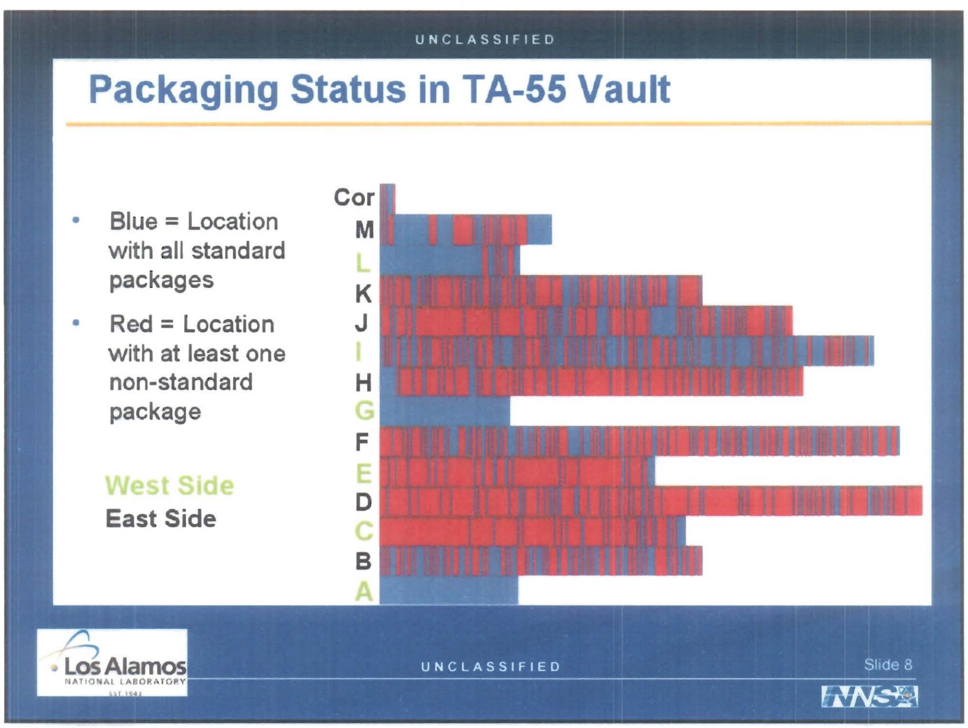
UNCLASSIFIED
Packaging Status in TA-55 Vault
Blue = Location with all standard packages
Red = Location with at least one non-standard package
West Side
East Side
Cor
M
L
K
J
I
H
G
F
E
D
C
B
A
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Schedule

	FY04-06	FY07	FY08	FY09	FY10
Programmatic	48%	32%	7%	2%	0%
Excess (2000-1)	10%	8%	3%	1%	0%
Risk remaining at end of the FY	58%	40%	10%	3%	0%

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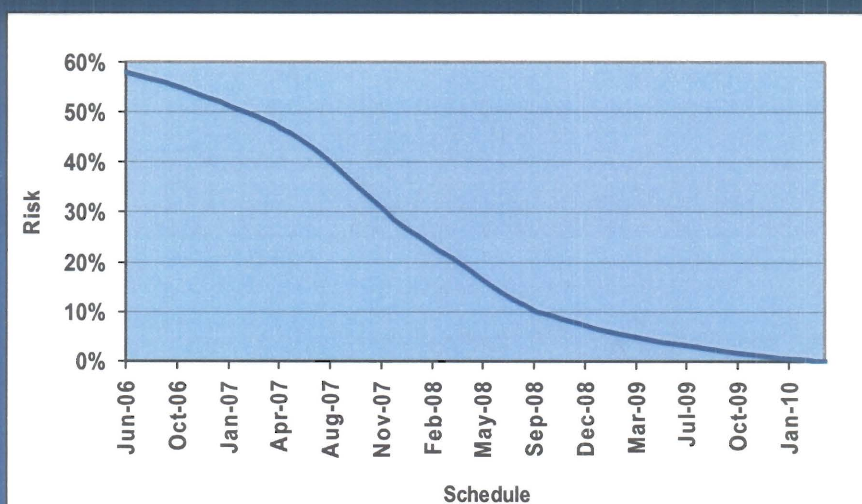
Risk Reduction Schedule



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Funding

	FY 2007 ($K)	FY 2008 ($K)	FY2009 ($K)
Pit	*1000	2000	2000
Heat Source	1500	200	N/A
RTBF	1800	2100	N/A
Container Quality Program	900	1000	700
Project Manage & Support	1500	1500	900
Total	6700	6800	3600

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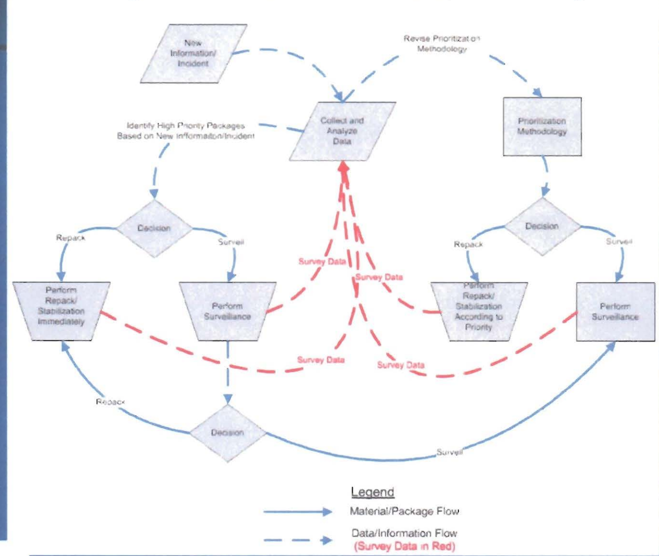
Integrated Surveillance and Disposition Logic



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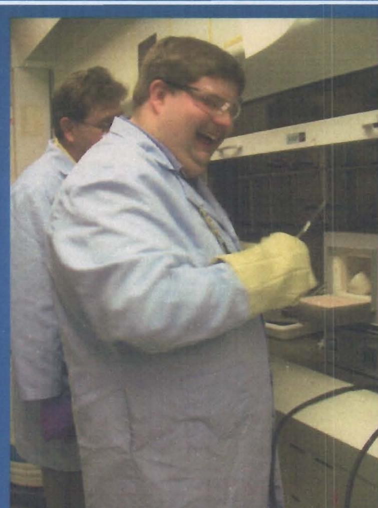
ISD Team Member



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NM Packaging Technical Review Board

- Enables and authorizes a team of TA-55 nuclear material packaging experts to review, provide input, evaluate feasibility and make recommendations to line management for approval of nuclear material storage packages for use at TA-55.

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Examples of NMPTRB Scope

- Systems Adequacy Analysis of Standard Nuclear Material Storage Containers (SNMC's) used at TA-55
- Evaluation of Procurement Specifications for New SNMC's
- Revisions of Nuclear Material Packaging and Storage Requirements Documents
- Review of process specific and/or unique nuclear material storage containers for use at TA-55.
- Evaluation of procedure changes that affect nuclear material storage containers and worker safety
- Advise on implementation of new DOE packaging and storage requirements
- Maintenance and surveillance requirements for nuclear packaging

NMPTRB Membership

- Respective group leaders nominate members. Co-chairs approve nominees and they are expected to serve up to a three-year term. Nominees should have expertise in one or more of the following areas:
  - Operational or user experience
  - Quality assurance, quality control, or performance surety
  - Authorization basis
  - Packaging engineering
  - System engineering
  - Program/project management

Summary

- Work packages for repack/consolidation/disposition of RTBF-owned, "orphan", and uranium for FY 07 in place.
- Feed lists for vault work-off to processing groups have been developed using criteria for west side strategy.
- Cost/benefit work in progress to choose best path forward for vault recovery.

Surveillance of Containers at LANL

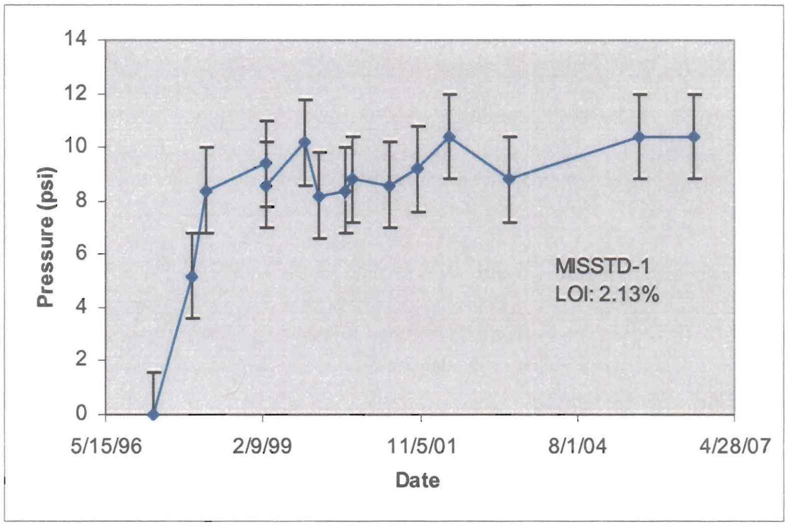
Surveillance & Monitoring Annual Meeting

January 23-25, 2007

Outline

- LOX/LM Container Examinations
- LANL packaged 3013 containers
- RFETS Metal Can examination
- Can puncture system at LANL
- Future Tasks

MISSTD1 Pressure Data



LOX/LM Plans

- Photo document container condition as they are opened.
- MISSTD-1
  - Puncture
  - Gas Sample
  - Photo document container condition
  - Reuse powder material

Excess Plutonium packaged in 3013 containers

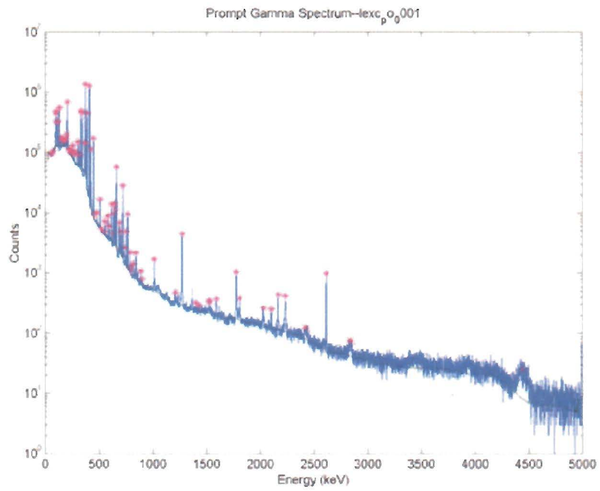
3013 inners loaded at Los Alamos - 40

- 12 Radiographs recently taken – Images are on the classified system
- Lid depth measurements taken

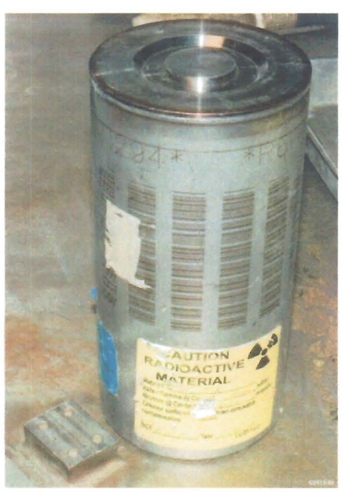
LEX/CP-001

- First 3013 Container Packaged at Los Alamos
  - Powder in convince can and inner 3013
  - Date packaged June 2001
- Net: 1684g
- SNM: 733 g (43.5 wt%)
- Composition: $MgCl_2$, NaCl, KCl
- Possible source: Cl ion exchange
- IDES: 217
- Radiograph on classified system.
  - Convince can intact
  - inner can not pressurized.
- Prompt gamma available

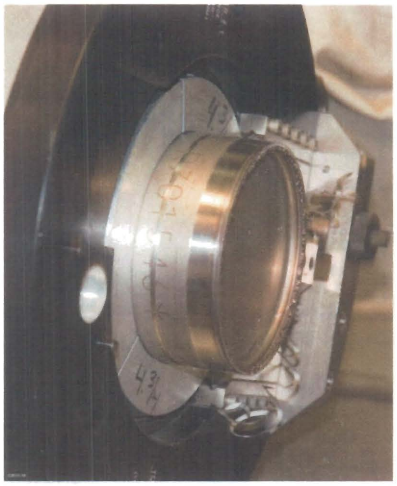
LEX/CP-001

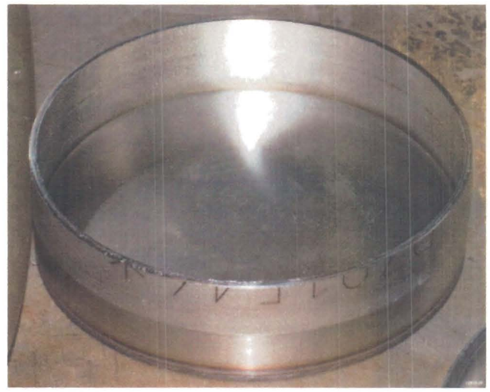


RFETS Metal Container at LANL



RFETS Metal Container at LANL
Lid removal

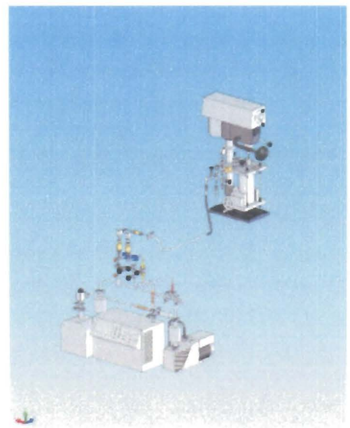




RFETS Metal Container at LANL

Can Puncture System

- Lid drilled thin enough to puncture
- Container punctured and gas collected
- Gas will be analyzed in room 105
- System will be operational in May



Surveillance Plans at LANL

- Open LOX/LM containers and photo document condition of inner and outer containers. Plan to open one per Week
- Open, gas sample and photo document MISSTD-1
- Open one or more LLNL containers, gas sample and photo document condition of inner and outer containers.
- Complete surveillance plan for 3013 containers packaged at Los Alamos

Plutonium Air Transport Container PAT-1 Modifications

Surveillance & Monitoring Annual Meeting
January 23-25, 2007

Outline

- Scope
- Proposed Container
- Technical Basis
  - Stainless Steel Baseline material
    - Eutectic Issue
      - Thermodynamics
      - Kinetics
      - Barriers to Thermodynamics and Kinetics
      - Sacrificial Materials
  - Alternate Materials
    - Eutectics
    - Corrosion
  - Packaging and Packaging Compatibility
- Summary
- Proposal

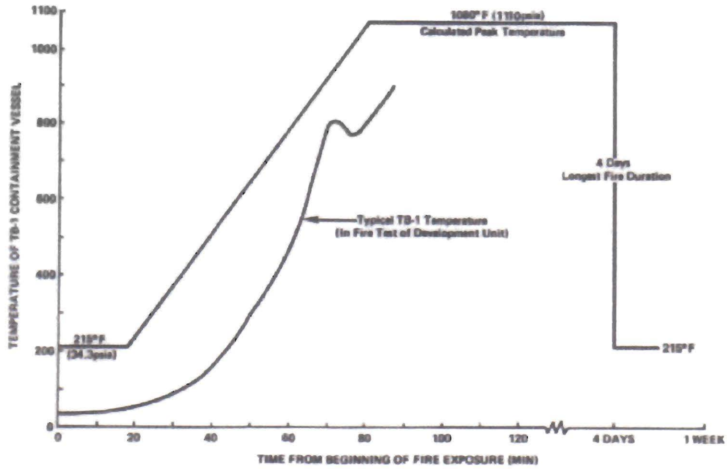
PAT-1 Current Certification

- Plutonium oxide and its daughter products in any solid form
- Mixtures of natural or depleted uranium oxide and plutonium oxide an their daughter products in any solid form
- Inner Container must weigh less than 2.1-kg

Material Scope

- The product container will hold
  - Small and large conflat containers holding hermeneutically sealed plutonium metal samples
  - Plutonium Metal – electro-refined metal ring broken into smaller pieces
  - Plutonium based neutron sources
- Container must safely contain plutonium metal
- Container must survive the design accident
- The product container must be as light as possible to allow the maxim payload

TB-1 Temperature/Pressure Profile



Transient Scope

- The container will never exceed 580C in a fire burning for 4 days
- Interactions will be evaluated at 850 C and below

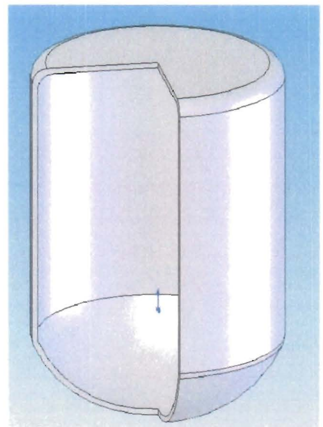
PAT-1

- Boundaries
  - PC-1 Product Can
    - Materials TBD
    - Hermetically sealed by weld or screw top 1rd lid
    - Wall thickness TBD
  - TB-1
    - PH13-8Mo stainless
    - hermetically sealed with copper gasket
    - Wall thickness 0.56-inches
  - Protective Over Pack 65-gallon drum with inner barrier
    - 304 stainless steel
    - Wood
    - steel

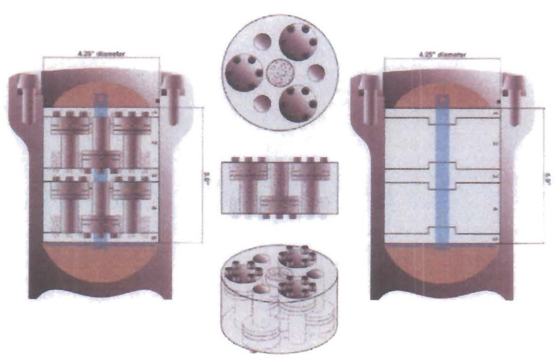
TB-1 System

PC-1 System – Design

- COMPATIBILITY BARRIER
- Curved bottom or not!
- Closure weld or Screw cap
- What Convince cans are allowed



PC-1
Inner Container Configuration



PuFe Phase Diagram

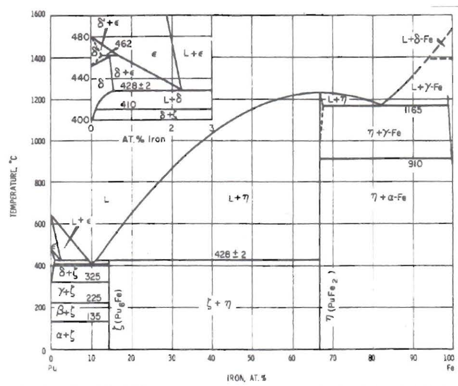


Fig. 7.11—Plutonium-Iron. From F. W. Schonfeld, in A. S. Coffinberry and W. N. Miner (Eds.), *The Metal Plutonium*, p. 243, The University of Chicago Press, Chicago, 1961.

Transient Solution

- Plutonium metal will never be in direct contact with stainless steel
- An Eutectic will never form until after plutonium melts which is a temperature greater than 640 C

Fe/Pu Position

- Unpublished Pu-Fe temperature tests have been made under ideal conditions, that is, with burnished plutonium and steel surfaces contacted under weight and surrounded by inert gas. Indirect references to the test results imply that under these ideal conditions, significant eutectic formation does not occur until temperatures reach 600°C.
- Below the Pu melting temperature the two metals have to be in intimate contact and that is unlikely because both quickly form an oxide layer that separates the two metals
- Conclusion: The eutectic is not a problem until one of the metals melts or at temperatures greater than 600°C for Pu metal

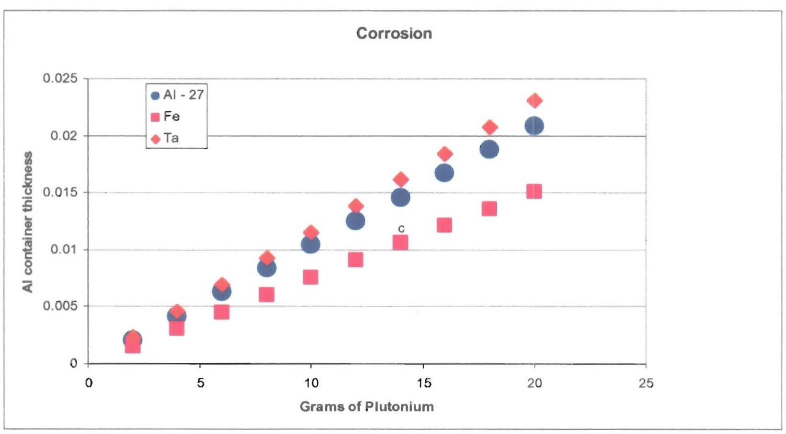
Technical Basis

- Potential Materials
  - Fe/Pu Eutectic Issues
  - Al
  - Ti
  - Ta
  - Nb
  - Zr
  - Mo
- Compatibility
- Radiological Considerations

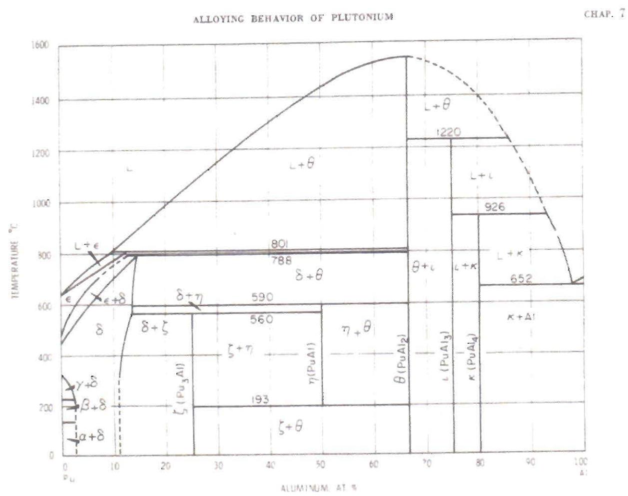
Potential Container Materials

Element	Eutectic or Peritectic Phase	Issue	Intermediate compound	Element or compound melt Temp. C
Al	Peritectic 10%@801C	Oxidizes at high Temperatures	PuAl2	About1550C
Ta	None	Oxidizes at high Temperatures	None	2996
Ti	None (Peritectic 20%@770C)	Oxidizes at high Temperatures		
Zr	None (solid solution)	Oxidizes at high Temperatures	None	1855
Nb	None	When processed at even moderate temperatures niobium must be placed in a protective atmosphere. oxidizes in air at 200 ° C	None	2477
Mo	1%/590C	Oxide evaporates	None	2617
W	None	Difficult to machine	None	3407
Fe	10%/410C	Potential eutectic melt failure	PuFe2	1240+5

Containers as Sacrificial Containers



Pu-Al Phases



Aluminum as a Container Material

- Pro's
  - Light weight
  - Plutonium – Addition of aluminum does not form a lower melting eutectic
  - Inexpensive
- Con's
  - Low melting temperature (660 C)
  - Forms a eutectic with iron at 650 C and 0.9 %Fe
  - Difficult to weld

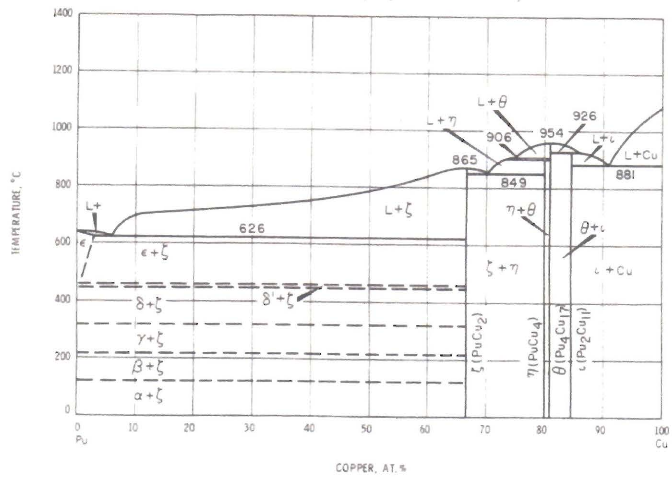
Round Robin Samples
Container and Samples

- Stainless steel sample containers
  - Plutonium Metal wrapped in tantalum foil
  - Package placed in Stainless steel container
- Stainless steel sample containers bagged out.
- Copper gasket hermetically seals the container
- Stainless steel lid

Space Filler

- Pieces projected from one end of the container to the other may damage the container
- Solution:
  - Metals
    - Fill empty spaces with aluminum honeycomb
  - Sample containers
    - Aluminum beads

Pu-Cu Phases



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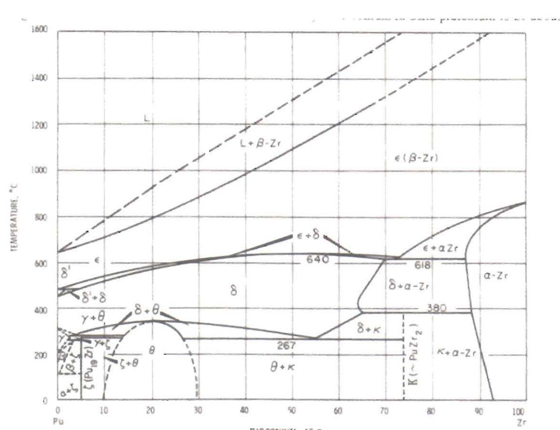
Pu-Zr Phases



Plutonium—Zirconium.[81, 82]

PC-1 System – Materials

- Plutonium Metal or Sample Containers (SS)
- Tantalum foil wrapped around sample
- Sample Holder – ConFlat Container
  - Stainless Steel
  - ID 0.875" or 0.5275"
  - Height 1.5-inches
  - Stainless Steel
  - Wall thickness?
- Bag out bag
- Aluminum honeycomb or alumina or aluminum beads
- PC-1
  - Container Material
    - Aluminum
    - tantalum
    - stainless steel
  - Screw top lid desired.
  - Wall thickness
    - TBD

Large Scale Shelf Life Surveillance

John Berg, Alex Carrillo, Max Martinez, Adam Montoya, Dennis Padilla, Kirk Veirs, Laura Worl (PMT-1)
David Harradine, Rhonda McInroy (C-PCS)
Scott Lillard (MST-6), Dave Kolman (NMT-15)
Jeremy Mitchell, Tracy Lee, Dave Rademacher (MST-16)

Surveillance and Monitoring Program Review Meeting
January 23-25, 2007
Savannah River Site, SC

NNSA — Los Alamos

Topical update

- **Review of results from PMAXBSC, the material in the corroded, leaking large-scale test.**
- **Gas generation from Master Blend material in large-scale corrosion tests.**
- **Corrosion results to date.**

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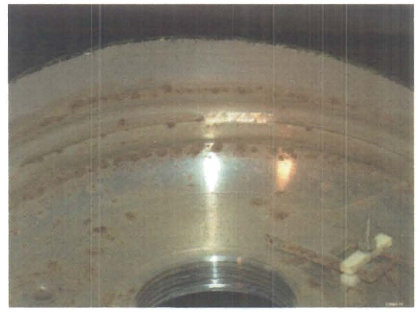
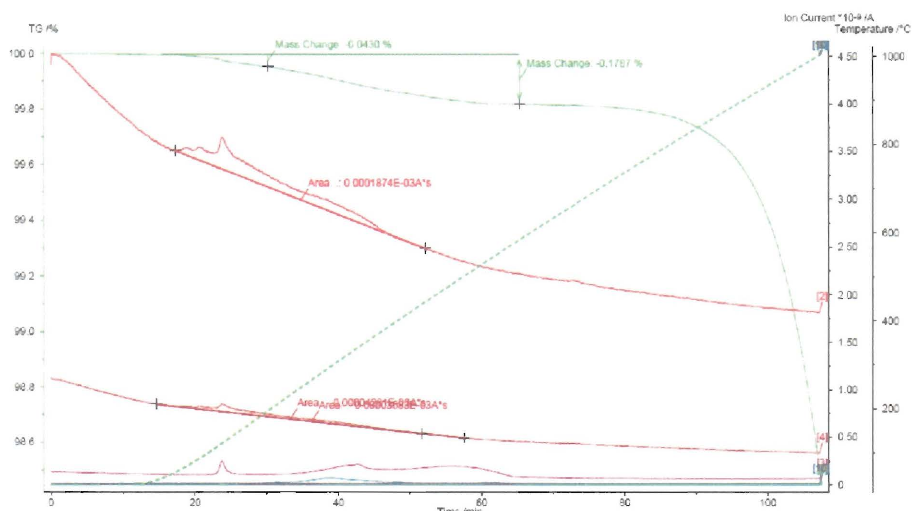
PMAXBSC

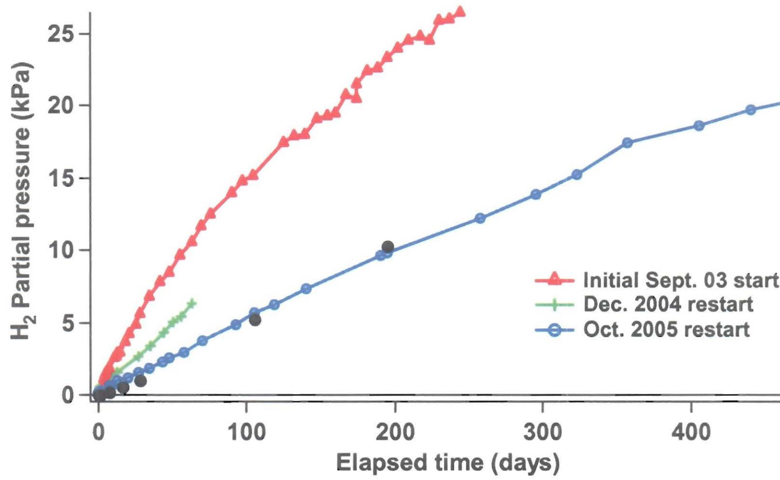


- Material
  - 800 deg C stabilization
  - 6-7% Cl, 2%Na, 4-5%K, 0.2% Mg
- Surveillance history
  - 0.2 wt% H_2O added by flowing humidified He.
  - 250 days surveillance in original container.
  - Paused to add 0.05% more H_2O
  - Confirmed leak and opened original container.
  - Apparent corrosion observed and leak path examined.
  - Material re-loaded in a new container for additional surveillance (400+ days).

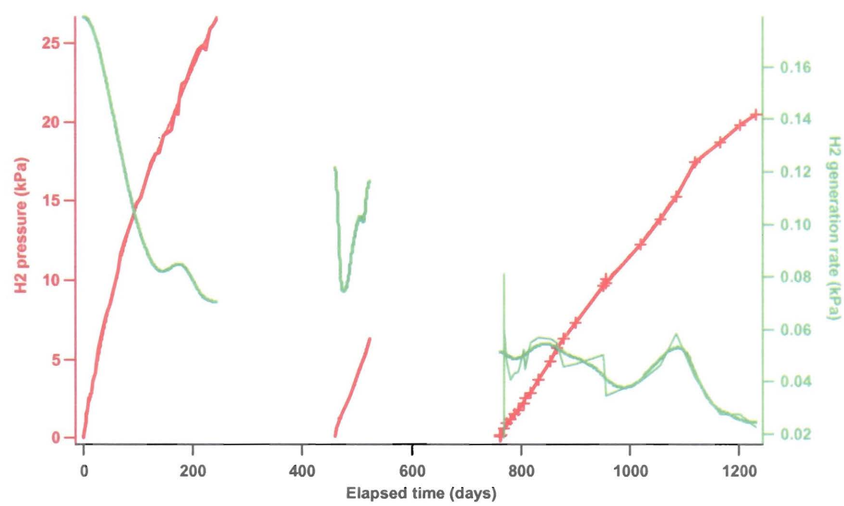
TGA-MS of PMAXBSC sampled before moisture addition



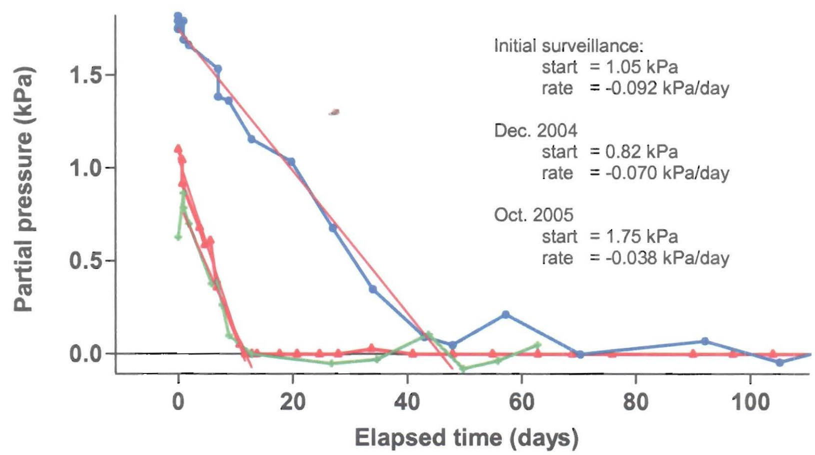
PMAXBSC - the material that originally showed corrosion of large-scale container in 2005.



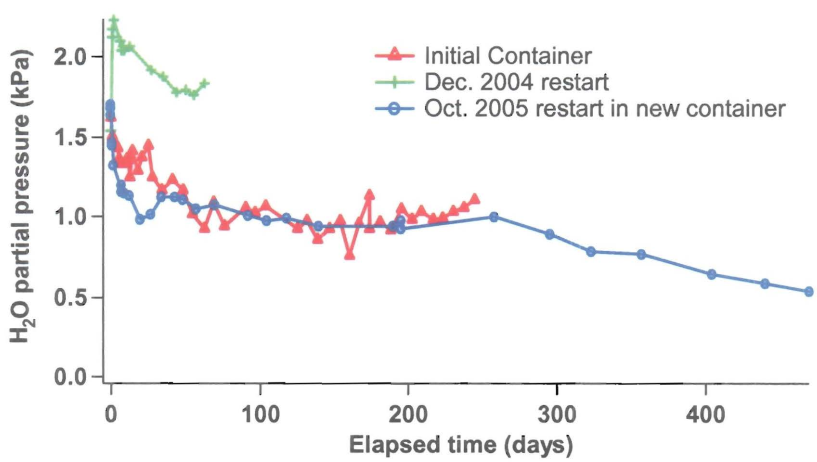
H_2 data from all PMAXBSC studies



PMAXBSC:
O_2 in the initial gas fill show linear depletion to zero



PMAXBSC:
H_2O in vapor phase decreases but remains detectable



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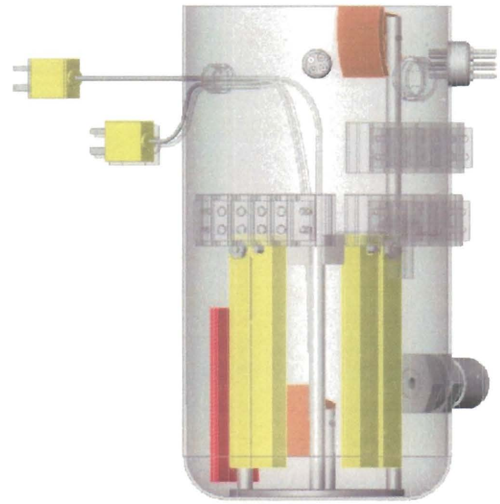
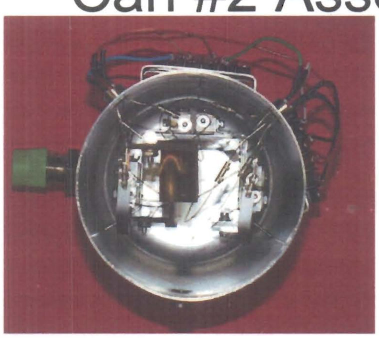
Container with corrosion samples, DCB specimens in material phase



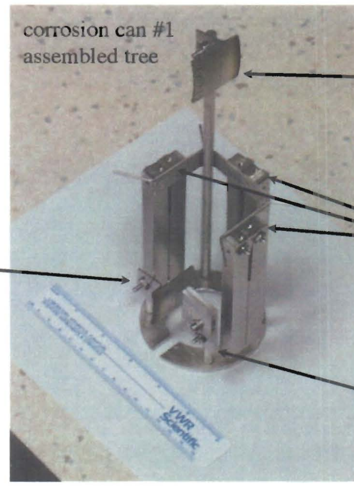
Can #2 Assembled, Pre-lid



top down

side

Can #1 Sample Tree



lid section, gas phase

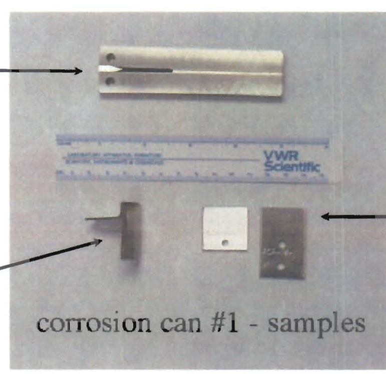
wedge loaded DCB specimens

lid section, contact region

artificial crevice assembly, contact region

Can #1 Samples

wedge loaded DCB



artificial crevice assembly

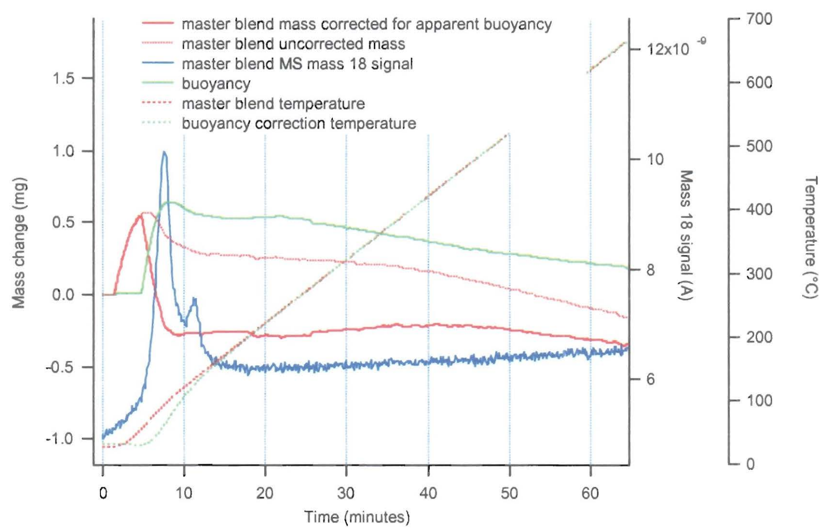
lid section

Master Blend Material

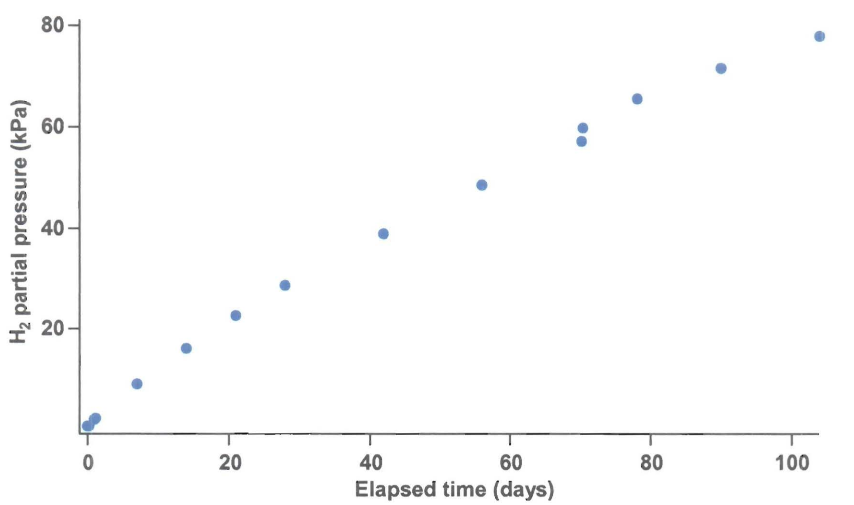
– 800 deg C stabilization
– ~12% Cl, 4%Na, 7%K, 0.6% Mg

Post stabilization

TGA analysis of Master Blend, ~ 0.1 % H_2O



Corrosion Can	K1scc DCB Samples (Quantity / stainless type in location)	Crevice Corrosion 3013 Lid Coupon Weld Samples (Quantity / stainless type / location)	Oxide Material	Load FY/ duration	Can Removal and DE
CAN 4B	ACRM samples	none	PMAXB SC	FY2005	FY2009
1	3 316L in material stress	- 1 inner LLNL / 316L / gas - 1 inner LLNL / 316L / material	Master Blend A	FY2006, 3 months	FY2006
2	3 316L in gas	- 1 inner SRS / 304L / gas - 1 inner SRS / 304L / material	Master Blend A	FY2006, 3 months	FY2006
3	3 316L in material crack tip chemistry examined	- 1 inner Han. / 304L / gas - 1 inner Han. / 304L / material	Master Blend A	FY2006, 3 months	FY2006
4	3 304L in material stress	- 1 inner RFETS. / 316L / gas - 1 inner RFETS / 316L / material	Master Blend A	FY2006, 3 months	FY2006
5	3 316L / TBD	- 1 inner LANL / 316L / gas - 1 inner LANL / 316L / material	Master Blend A	FY2006, long term	FY2008
6	tbd based on results	- 1 outer BNFL laser / gas - 1 outer BNFL laser / material	tbd	FY2006, long term	FY2008
7	Tbd based on results	- 1 outer BNFL GTAW / gas - 1 outer BNFL GTAW / material	tbd	FY2006, long term	FY2008



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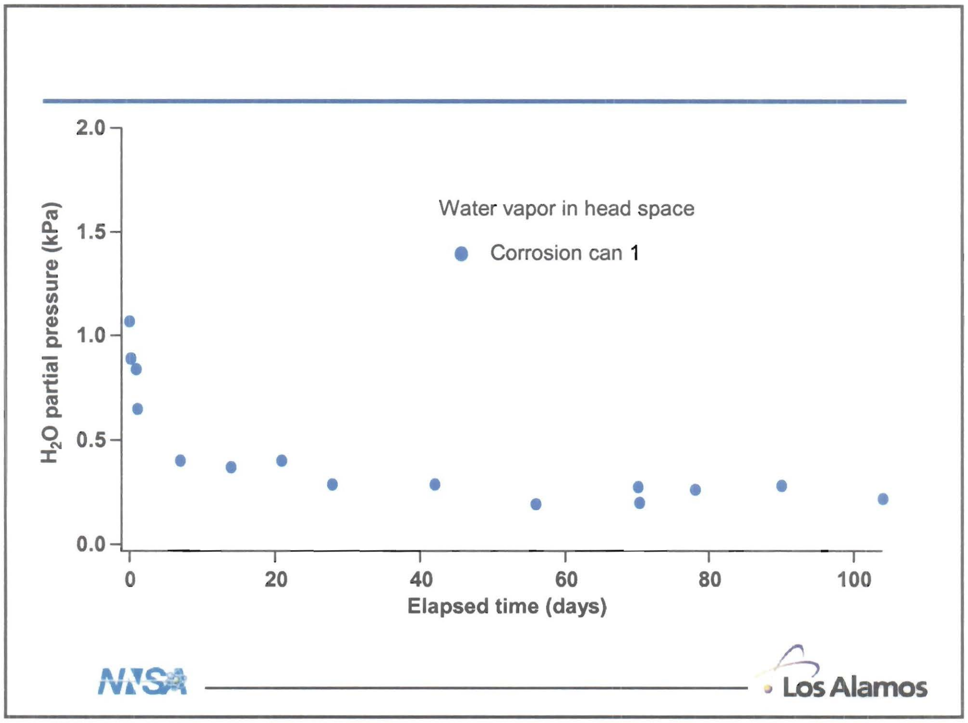
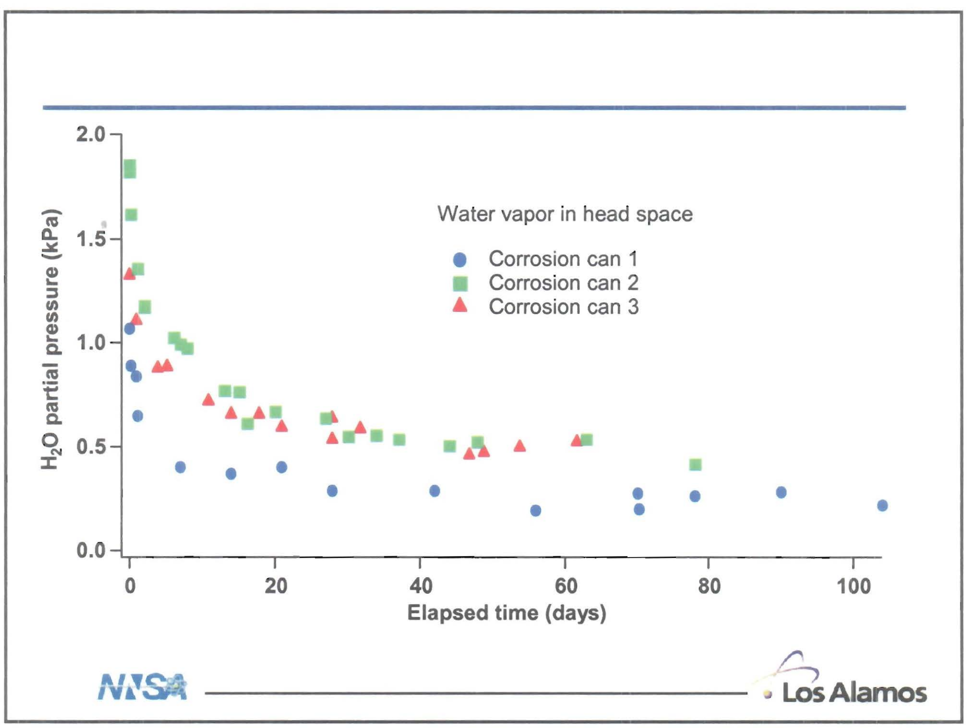
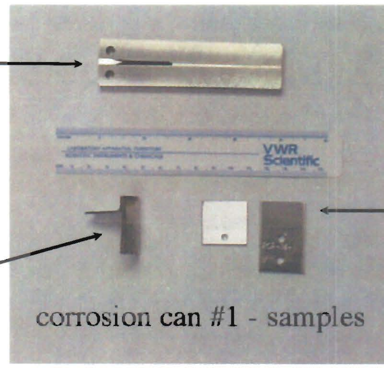


2.0
1.5
1.0
0.5
0.0
H_2O partial pressure (kPa)
Water vapor in head space
Corrosion can 1
0
20
40
60
80
100
Elapsed time (days)
NNSA
Los Alamos



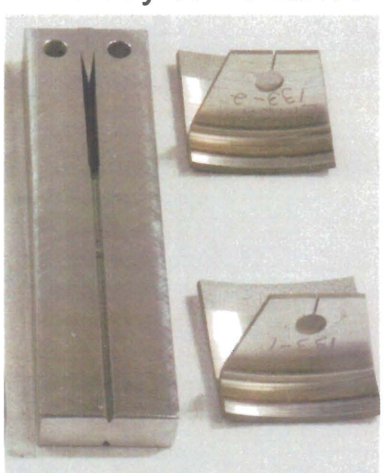
2.0
1.5
1.0
0.5
0.0
H_2O partial pressure (kPa)
Water vapor in head space
Corrosion can 1
Corrosion can 2
Corrosion can 3
0
20
40
60
80
100
Elapsed time (days)
NNSA
Los Alamos

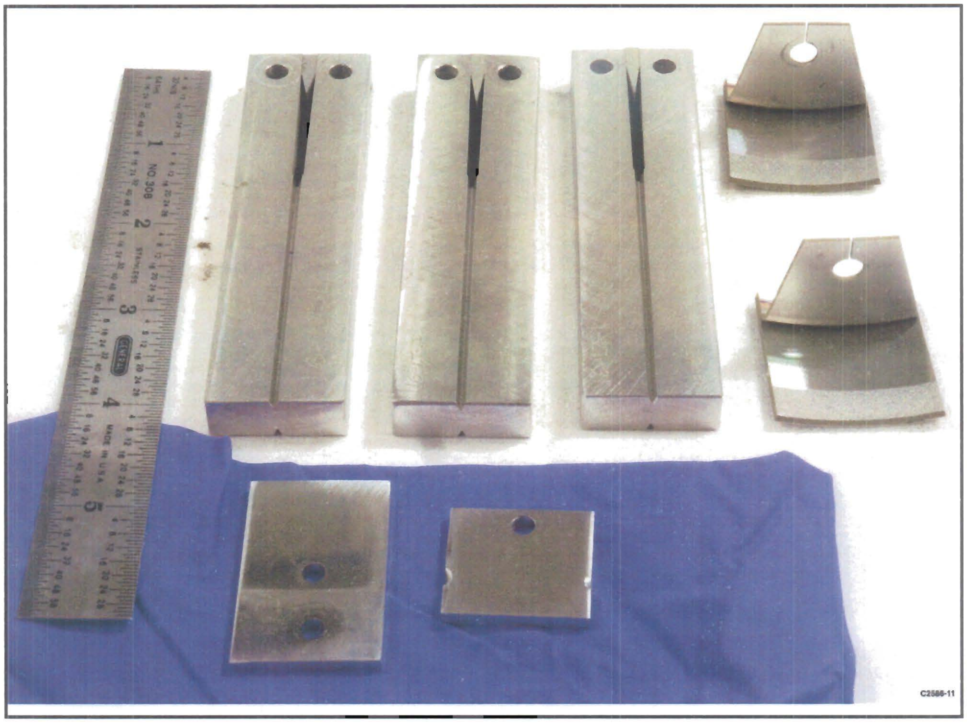
Can #1 Samples



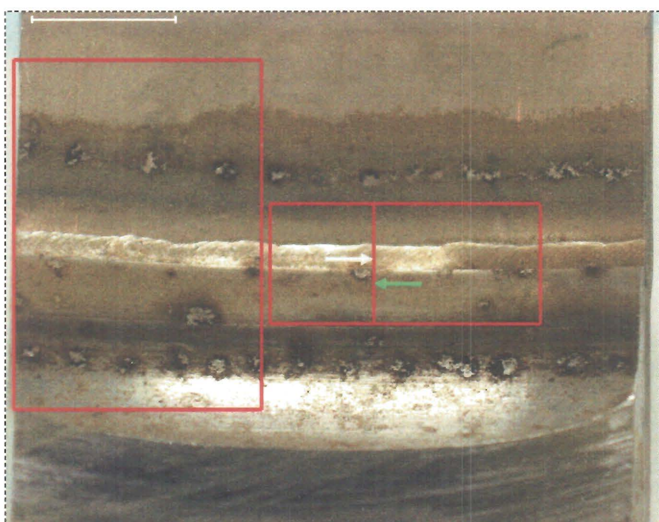
Corrosion Can 1

Visual inspection after 105 day surveillance





Can 4b Lid, Sectioning Layout



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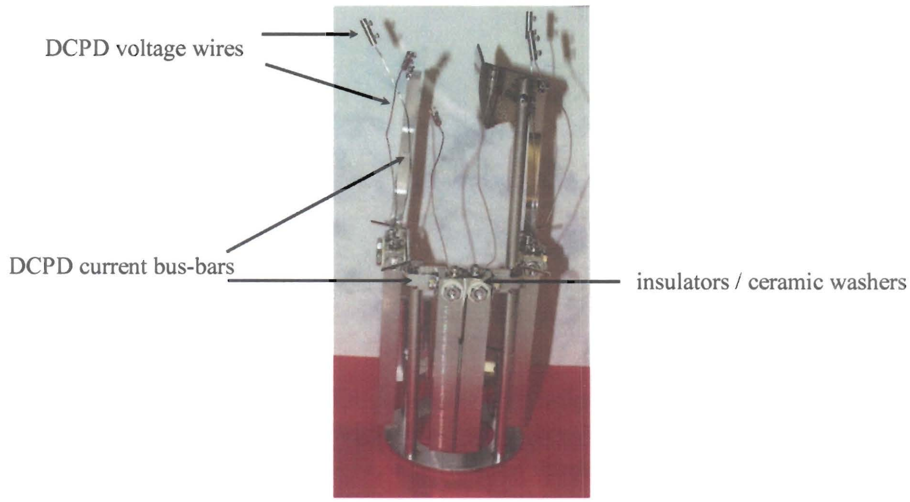
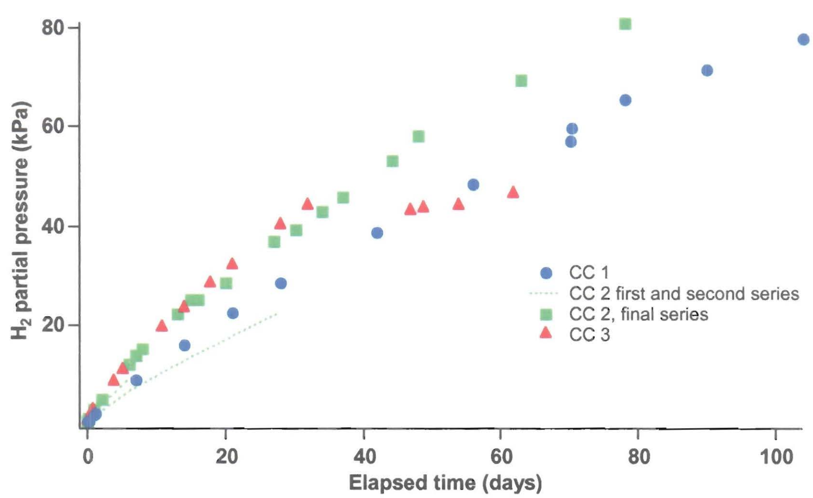
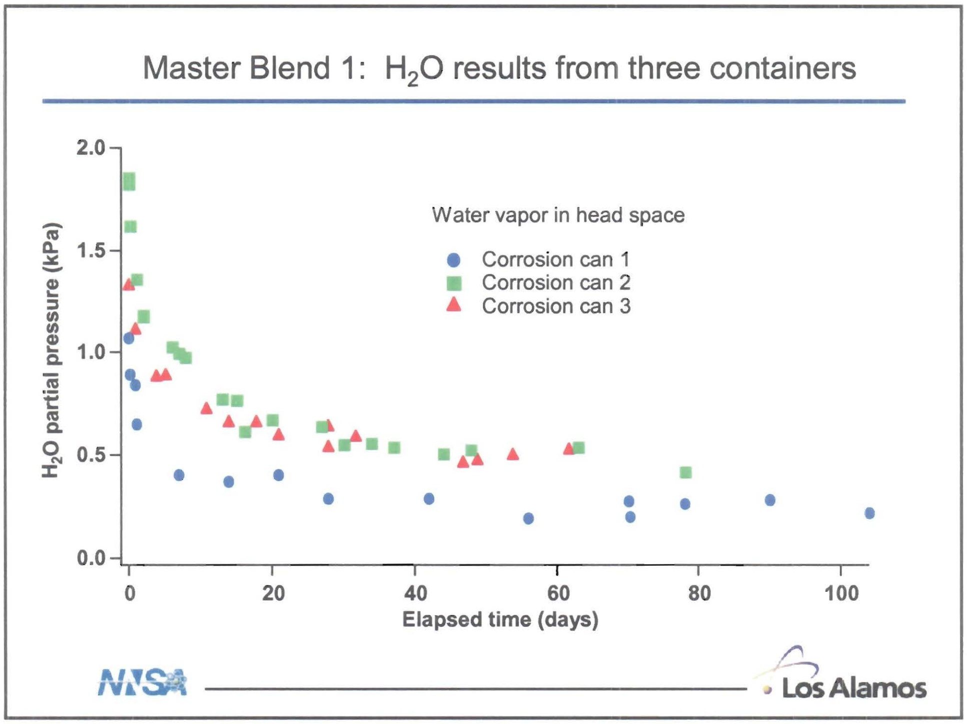
Can #2 Sample Tree

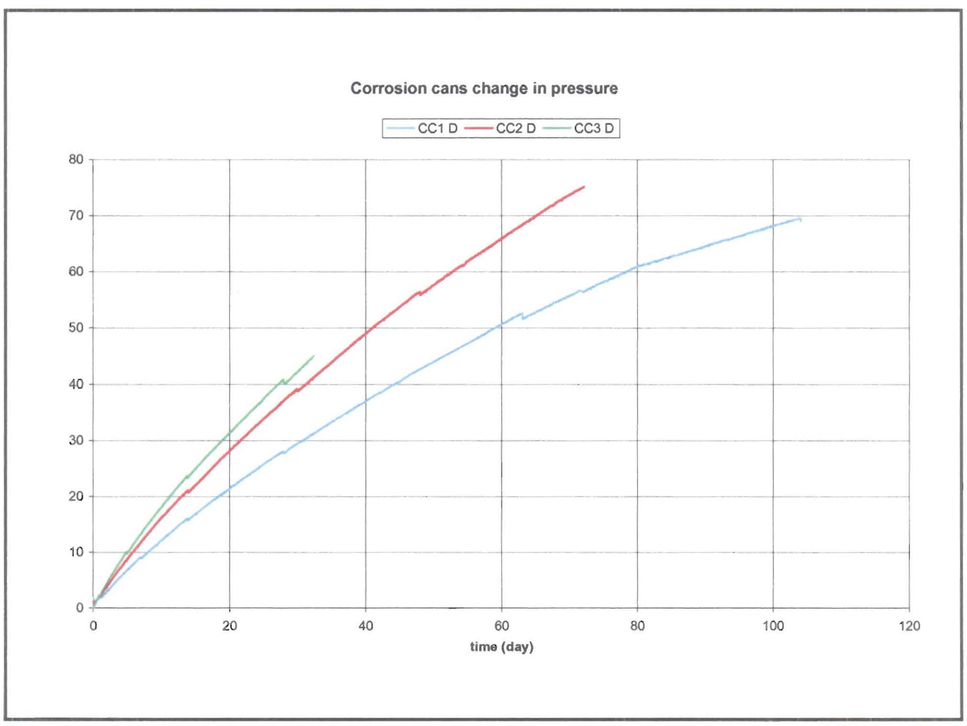


Master Blend 1: H_2 results from three containers

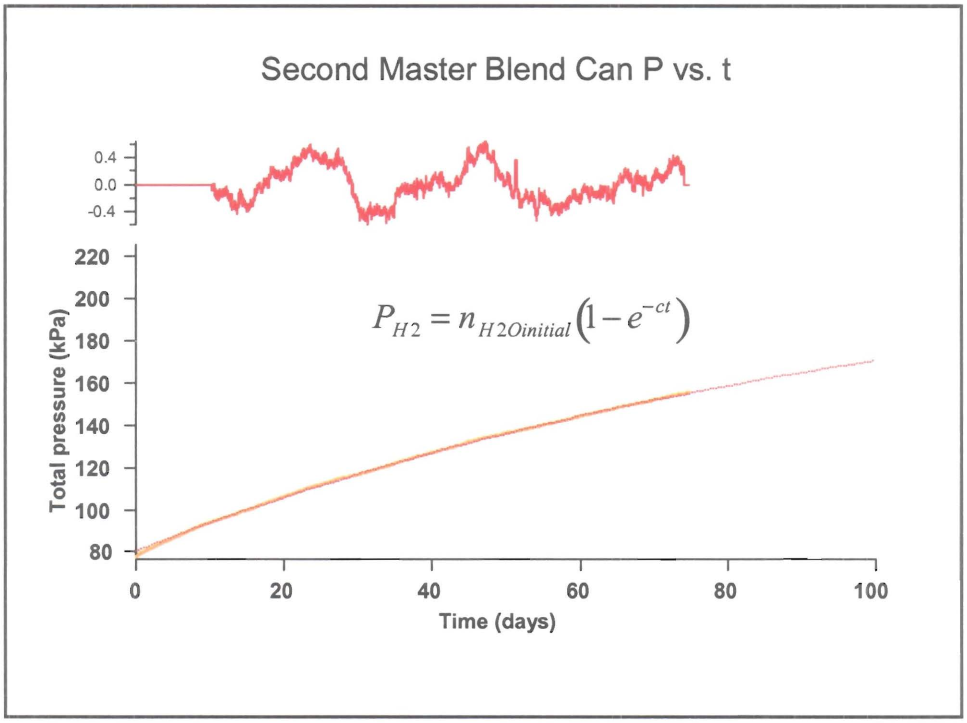


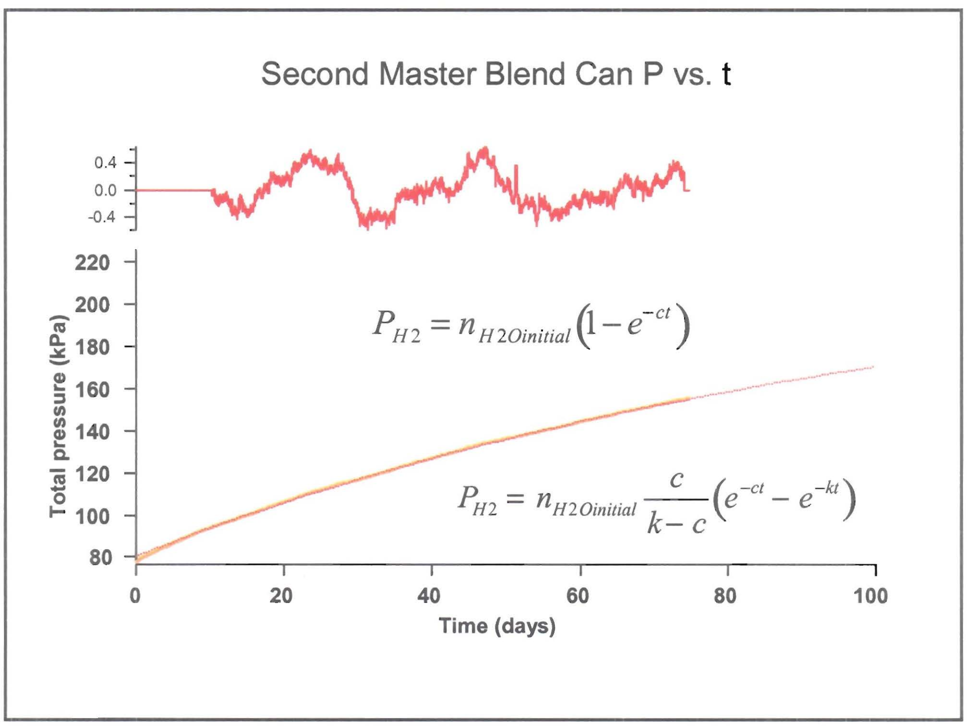


Master Blend 1: H_2O results from three containers
Water vapor in head space
Corrosion can 1
Corrosion can 2
Corrosion can 3
H_2O partial pressure (kPa)
2.0
1.5
1.0
0.5
0.0
0
20
40
60
80
100
Elapsed time (days)
NNSA
Los Alamos

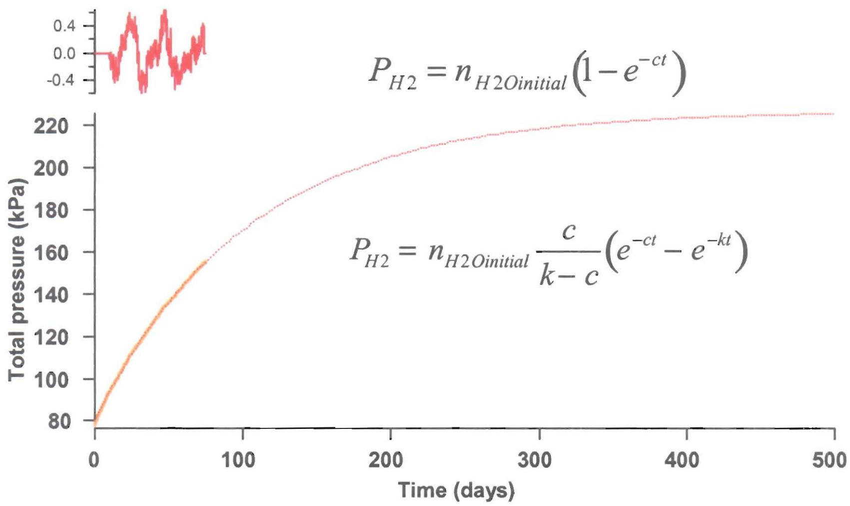


Corrosion cans change in pressure
CC1 D
CC2 D
CC3 D
80
70
60
50
40
30
20
10
0
0
20
40
60
80
100
120
time (day)


Second Master Blend Can P vs. t
0.4
0.0
-0.4
220
200
180
160
140
120
100
80
Total pressure (kPa)
$P_{H2} = n_{H2Oinitial}\left(1 - e^{-ct}\right)$
0
20
40
60
80
100
Time (days)

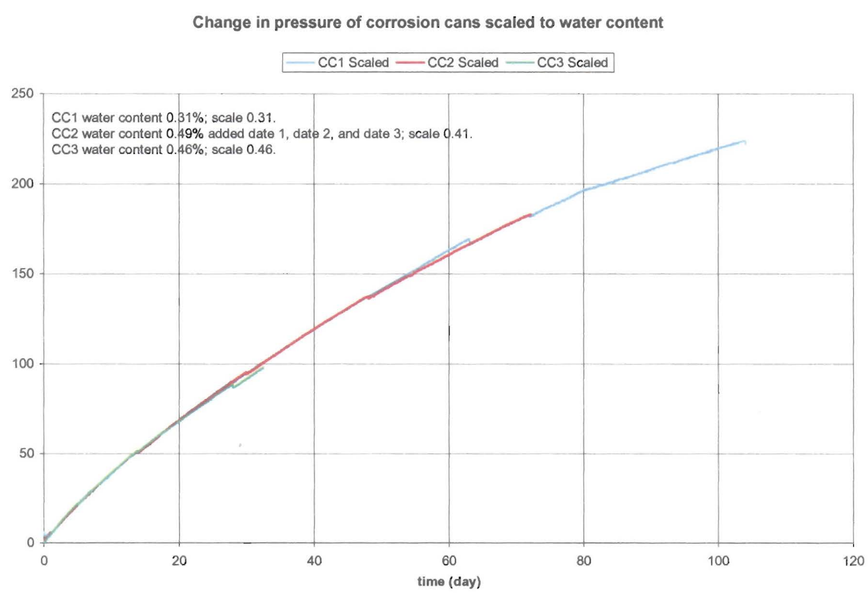

Second Master Blend Can P vs. t
0.4
0.0
-0.4
220
200
180
160
140
120
100
80
Total pressure (kPa)
$P_{H2} = n_{H2Oinitial}\left(1 - e^{-ct}\right)$
$P_{H2} = n_{H2Oinitial}\frac{c}{k-c}\left(e^{-ct} - e^{-kt}\right)$
0
20
40
60
80
100
Time (days)

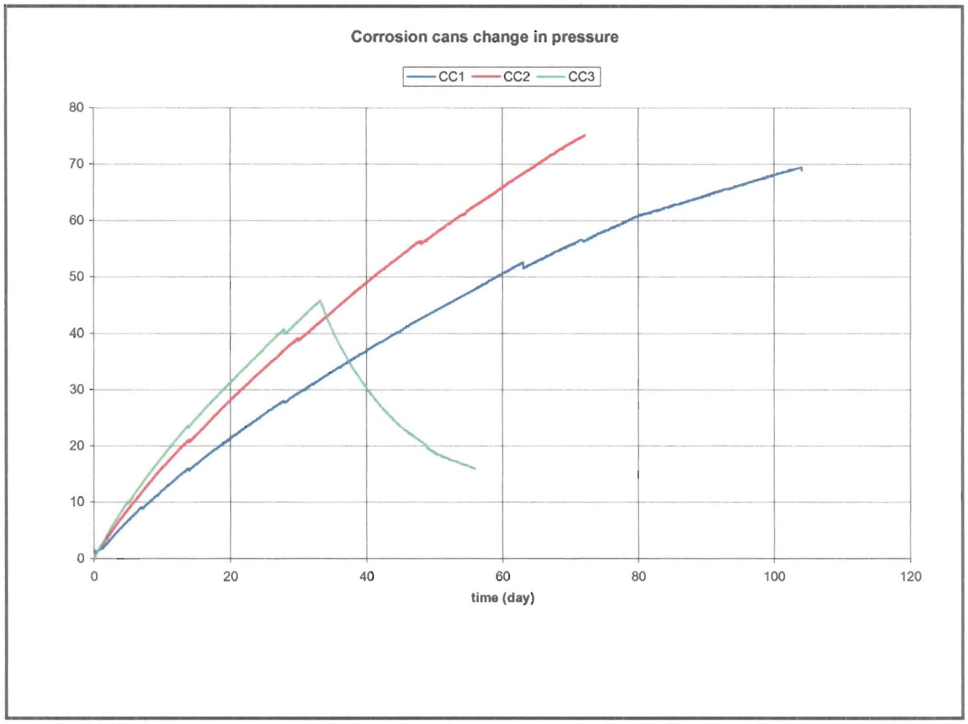
Second Master Blend Can P vs. t



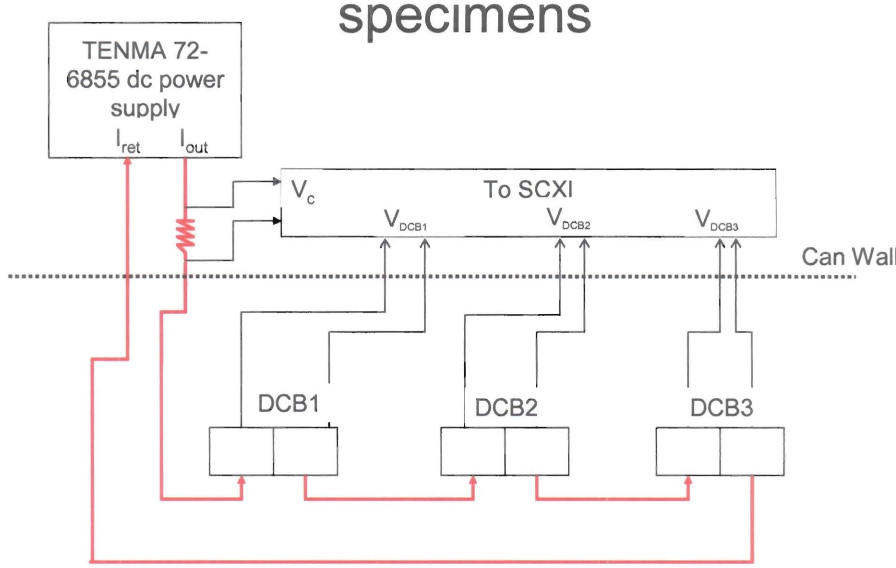
$$P_{H2} = n_{H2Oinitial}\left(1 - e^{-ct}\right)$$

$$P_{H2} = n_{H2Oinitial}\frac{c}{k-c}\left(e^{-ct} - e^{-kt}\right)$$

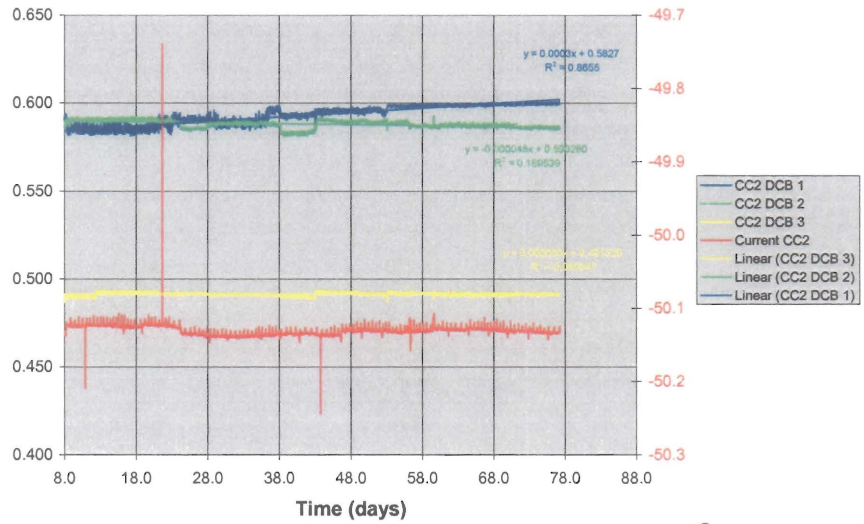




Electrical schematic for DCB specimens



CC2 DCB Crack Growth Monitoring Data



NNSA Los Alamos

PMAXBSC vs. Master Blend Comparison

	PMAXBSC	MB CC1	MB CC2,3
H_2O	0.2%	0.3 %	~0.45 %
Mg	0.2 % (0.016%)	0.6 % (0.28 %)	0.6 % (0.28 %)
Relative humidity at 30 days	35 %	< 10%	20 %
Initial H_2 gen. rate (kPa/day)	0.16 - 0.05	1.2	1.8
O_2 consumption rate (kPa/day)	0.1 - 0.04	>10	>10
Extrapolated H_2 (kPa)	100 - 300	150	170

NNSA Los Alamos

11589 Action Plan Overview

Strategy to understand the scope of potential O_2 generation indicated by small-scale shelf-life studies.

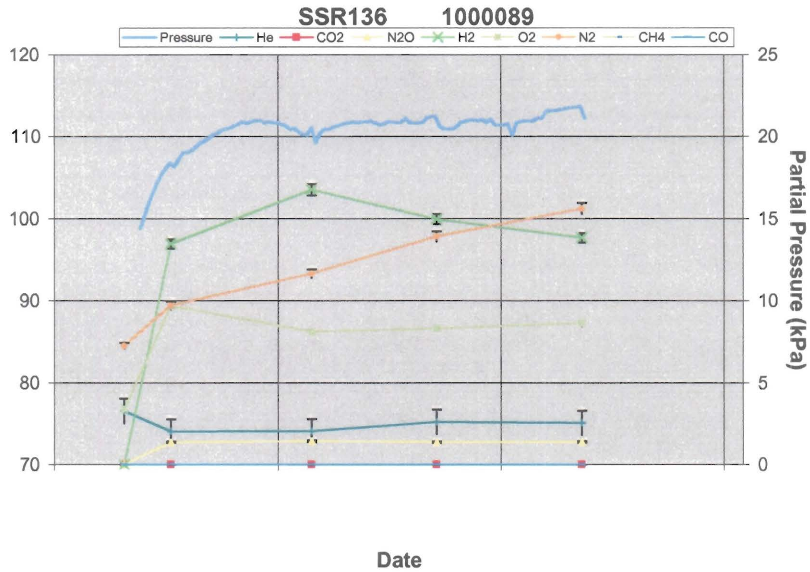
John Berg, Ed Garcia, Kirk Veirs

Concepts guiding the action plan

- Gas generation in at least some impure PuO_2 materials seems to be connected to alkaline earth chlorides ($MgCl_2$, $CaCl_2$) and related ternary alkali-alkaline earth salts that survive calcination and then are easily re-hydrated upon exposure to moist air.
- The water-soluble fraction of MIS material 11589 contains small amounts of both calcium and magnesium, along with chlorine, which is a indication that alkaline earth chlorides are present.
- 11589 has an abundance of soluble calcium relative to magnesium and relative to total chlorides, and a very low total amount of chlorides.

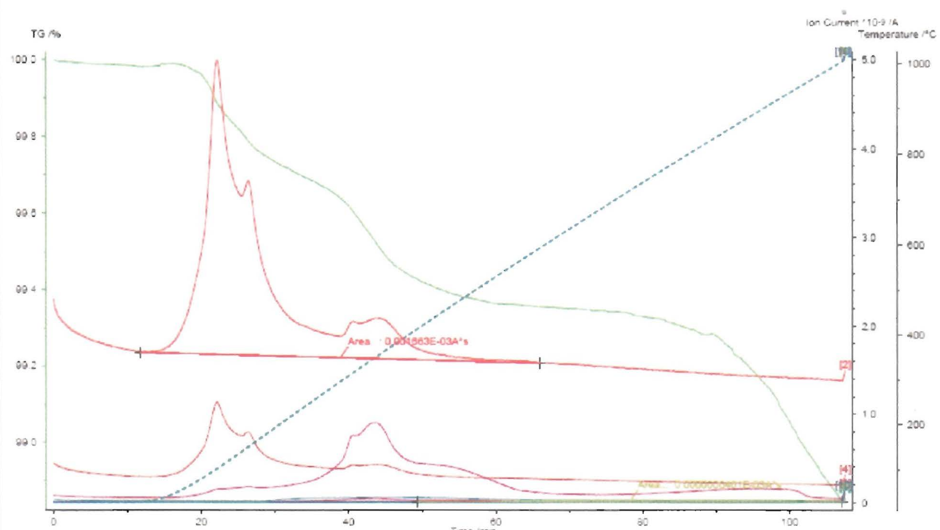
Proposed work (7/06)

- Gas generation studies on materials of known, controlled composition.
  - Pure PuO_2 (PEOF1) mixed with pure anhydrous $CaCl_2$
  - Pure PuO_2 (PEOF1) mixed with 11589 salt simulant
- Determine the dependence of 11589 gas generation on moisture content .
  - Confirm moisture content of 11589 in the shelf-life studies.
  - Repeat the shelf life study on 11589 material using at least one moisture content that is lower than the 0.5 mass % targeted in the current study.



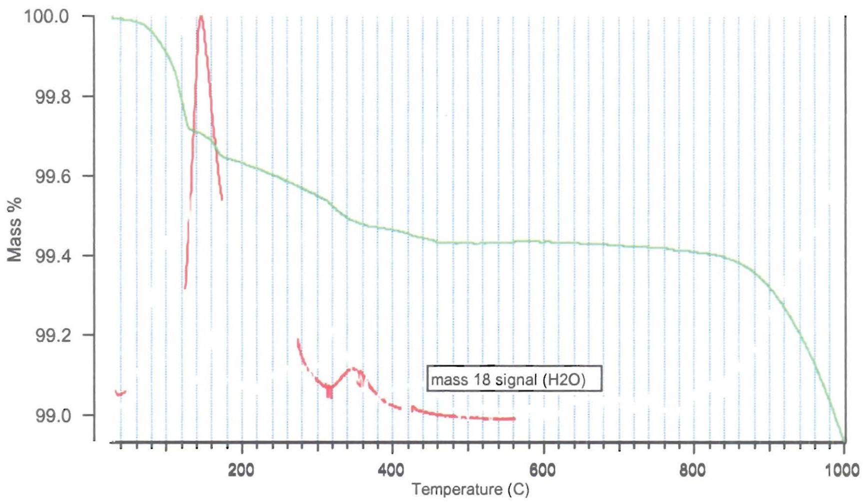
TGA-MS of 1000089 sampled before moisture addition.

Data collected by Tracy Lee, 1/18/07



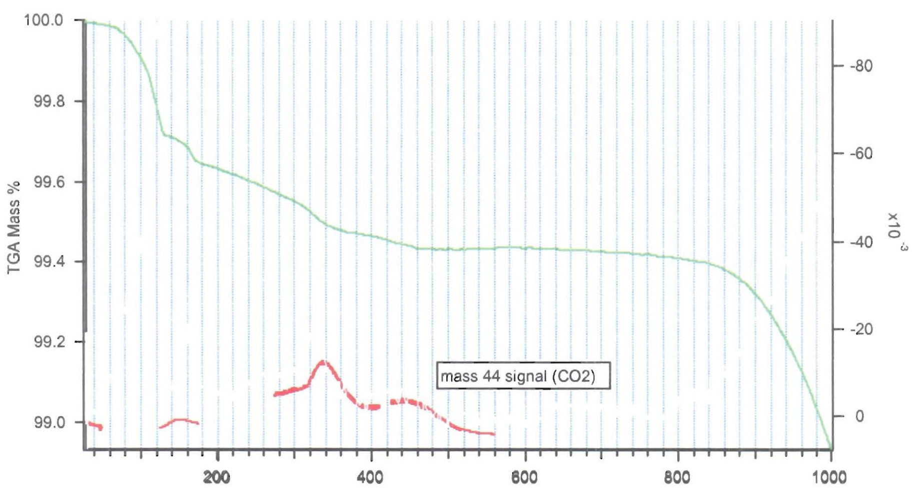
TGA-MS of 11589 sampled before moisture addition.

Data collected by Tracy Lee, 1/18/07 (aged 2.8 years)



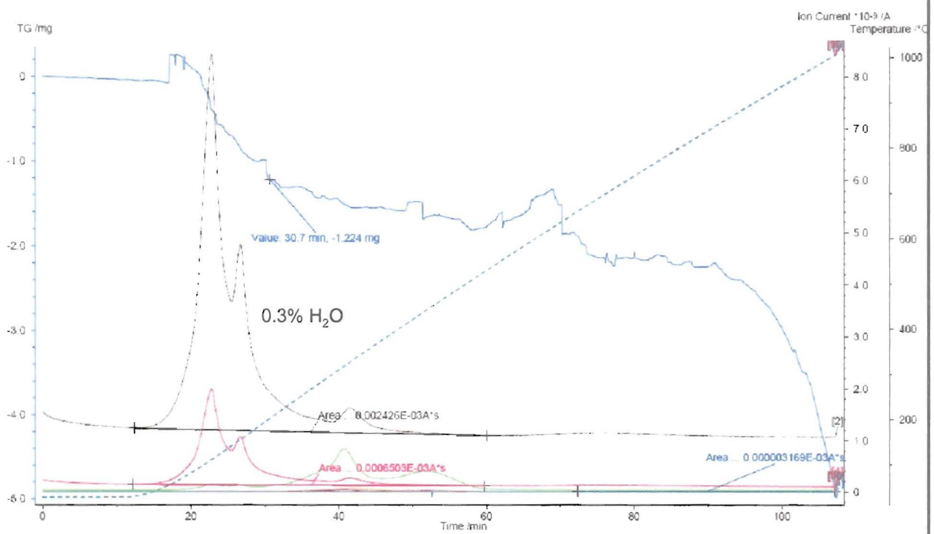
TGA-MS of 11589 sampled before moisture addition.

Data collected by Tracy Lee, 1/18/07 (aged 2.8 years)

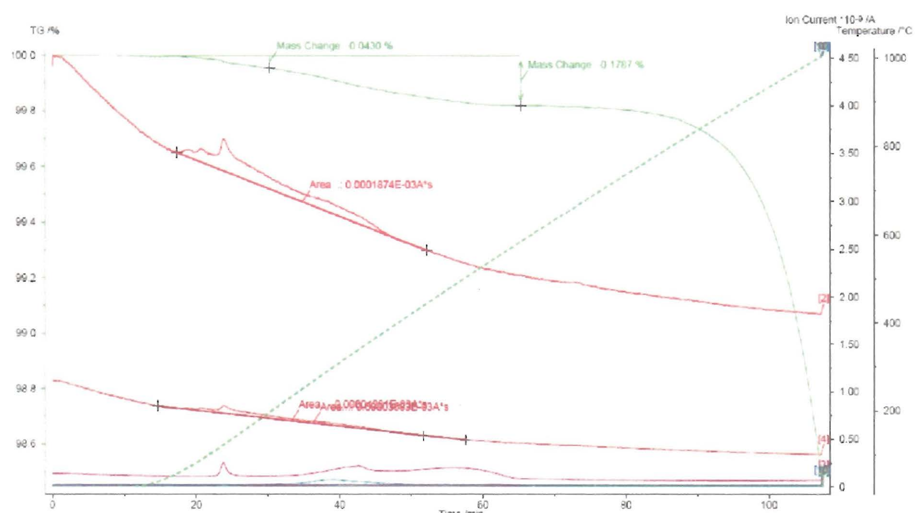


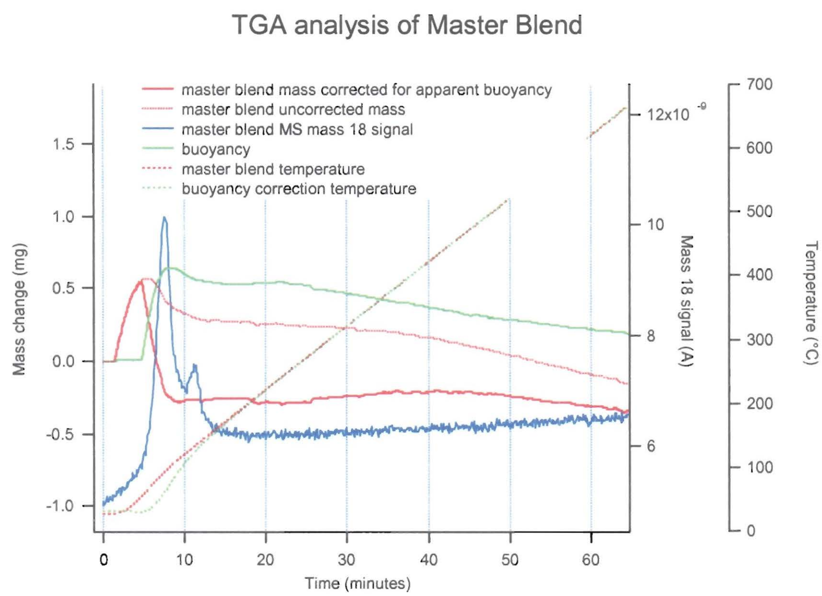
TGA-MS of 11589 sampled before moisture addition.

Data collected by Tracy Lee, 1/18/07



TGA-MS of PMAXBSC sampled before moisture addition



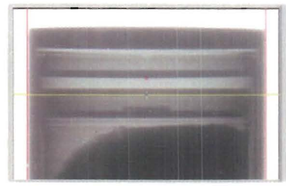


Status of proposed work (1/07)

- Gas generation studies on materials of known, controlled composition.
  - ✓ Pure PuO_2 (PEOF1) mixed with pure anhydrous $CaCl_2$
  - ✓ Pure PuO_2 (PEOF1) mixed with 11589 salt simulant
- Determine the dependence of 11589 gas generation on moisture content .
  - ✓ Confirm moisture content of 11589 in the shelf-life studies.
  - – Repeat the shelf life study on 11589 material using at least one moisture content that is lower than the 0.5 mass % targeted in the current study.

PFP Fiscal Year 2006 3013 Surveillance Activities, Including ISP Field Surveillance.

Presented to
The MIS Working Group
FY 06 Annual
MIS Meeting
Savannah River Site



T.J. Venetz
Fluor Hanford

S. E. Clarke
DOE/RL

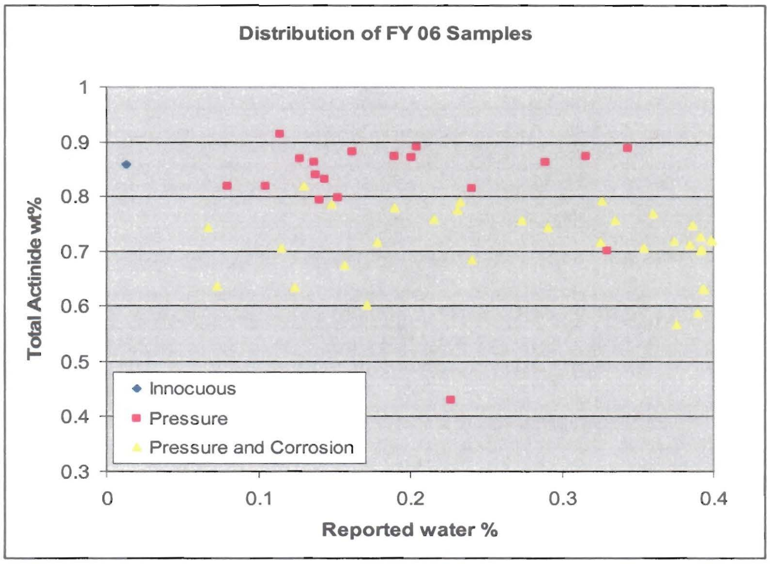
January 23-25, 2007

Fluor Hanford

Hanford FY 06 Samples

- **24 ISP samples selected by the Working Group**
  - 18 Randomly Selected
  - 6 By Engineering Judgment
    - Pure oxides, Rocky Flats Oxide (ARF), Oxide from impure solutions all with "high water"
- **29 additional PFP samples**
  - 13 items like MIS item 11589, with flammable gas issue
    - All those judged "most similar",
    - Those judged potentially similar with over 0.3 wt% water
  - 4 items with highest post sample weight gain
  - 6 ARF items with detectable weight gain or greater than 9 grams total water
  - Other Items processed in "wet" line with high reported water content
  - 4 repeats from FY 05 sample
- **Prompt Gamma Analysis on items judged "Potentially Similar" to MIS item 11589**

Sample Makeup

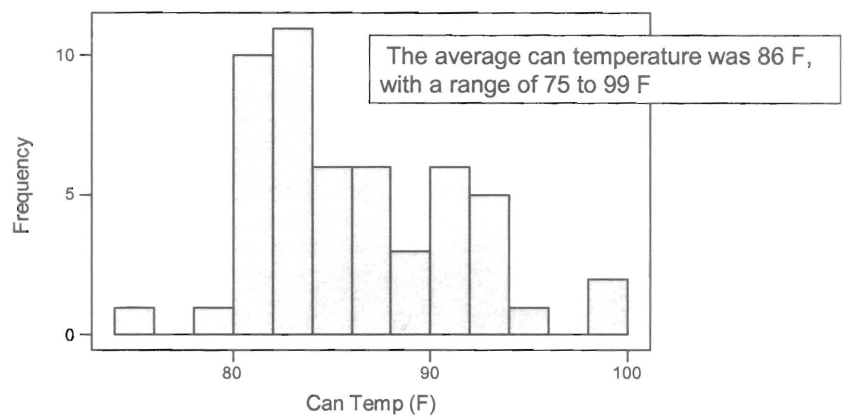


Results Obtained for

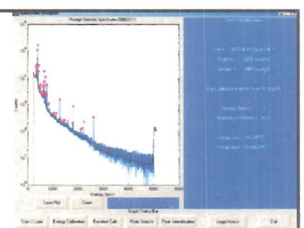
- Temperature (can and vault ambient)
- Contamination Status – All nondetectable
- Dose Rates
  - Individual maximums - 270 mr/hr gamma, 110 mr/hr neutron
- Visual Inspection – No abnormalities noted
- Gross Weight Change
  - All within 2 grams/none detectable
- Lid Deflection Change – follows
- Prompt Gamma Analysis - follows

Temperature Results

Histogram of Can Temperature (F)



Prompt Gamma Results



- **ISP and PFP Surveillance samples**
  - 28 items have Chloride, 1 has strong Fluoride
  - All were correctly assigned to Pressure and Corrosion Bin
  - One ARF item originally binned as Pressure and Corrosion shows only weak Fluoride
  - Pure Oxide and Mixed Oxide samples show either no impurities or only trace Impurities (Al, Mg, Na)
  - 2 ISP samples found to have anomalous PG spectra and have been rerun
- **Items Potentially Similar to 11589 (96 were identified for Hanford)**
  - Of 48 analyzed so far, only 5 are judged similar by Prompt Gamma
  - Prompt Gamma analysis still ongoing for the remaining 38 items

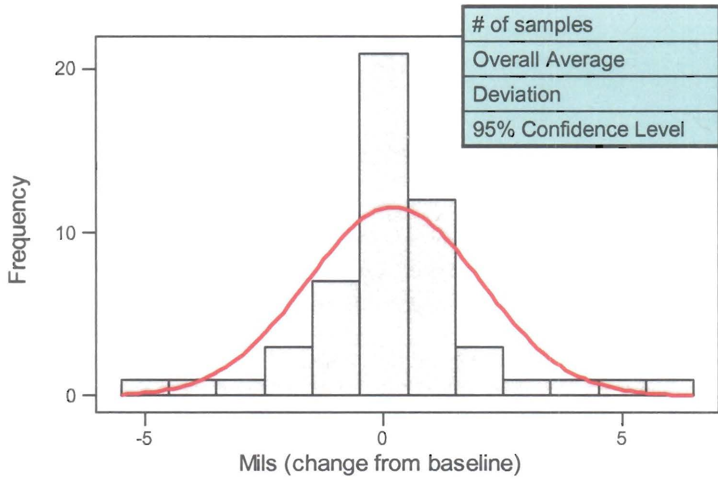
Radiography

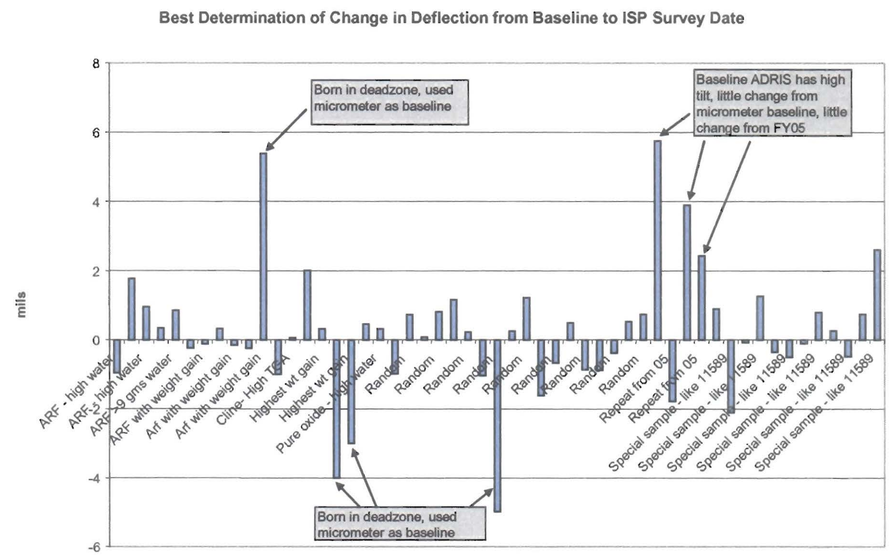
- **All containers inspected with new ADRIS ver 1.3 allowing multiple angle imaging to mitigate deadzone issue**
  - Traditional ADRIS – 4 angle average
  - Select View – pick best of existing 2 views - 4 angles
  - Best View – Pick best of 8 views – 16 angles
- **Requires careful evaluation off-line**
  - Writeup on best determination methodology for each can included in report on PFP Fiscal Year 2006 Surveillance Activities (HNF-31775) and in surveillance database

Radiography

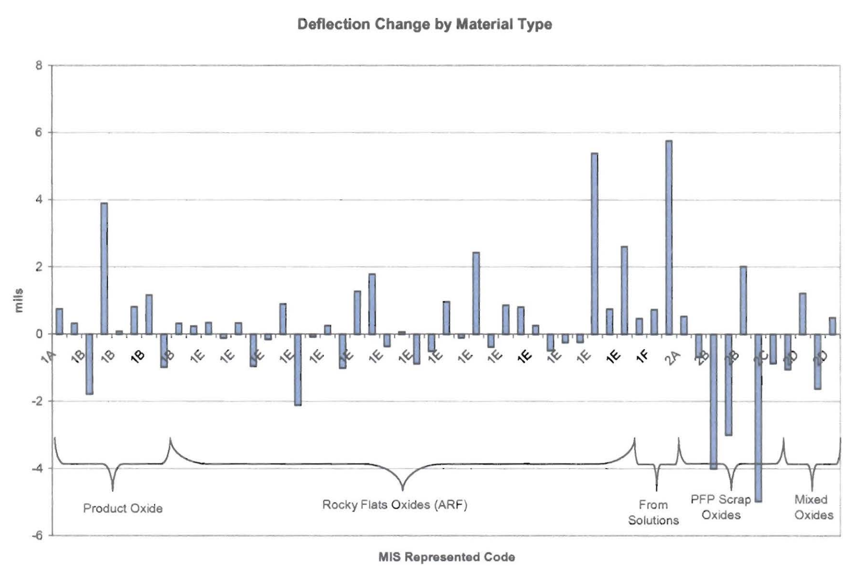
Histogram of Best Determination of Deflection Change -FY06

# of samples	53
Overall Average	+0.2
Deviation	1.8
95% Confidence Level	-0.3 to +0.7



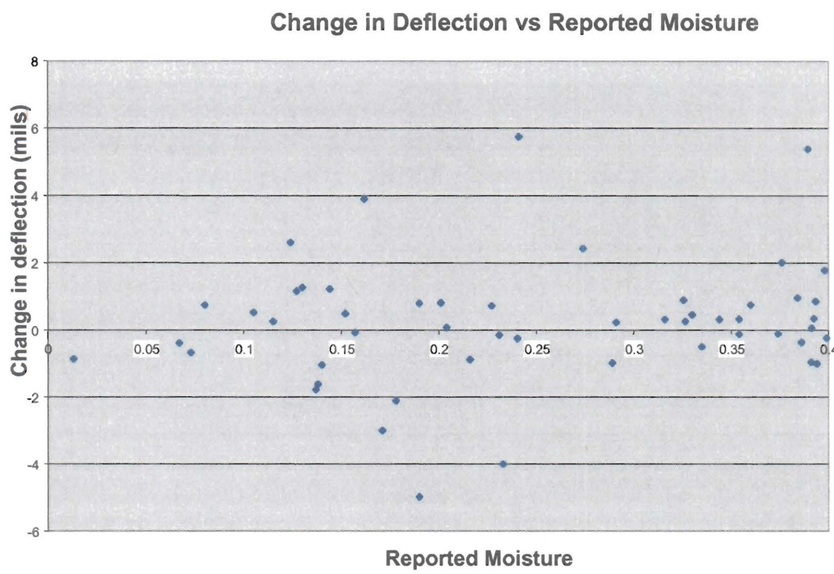


HNF-31944-VA Rev 0 9



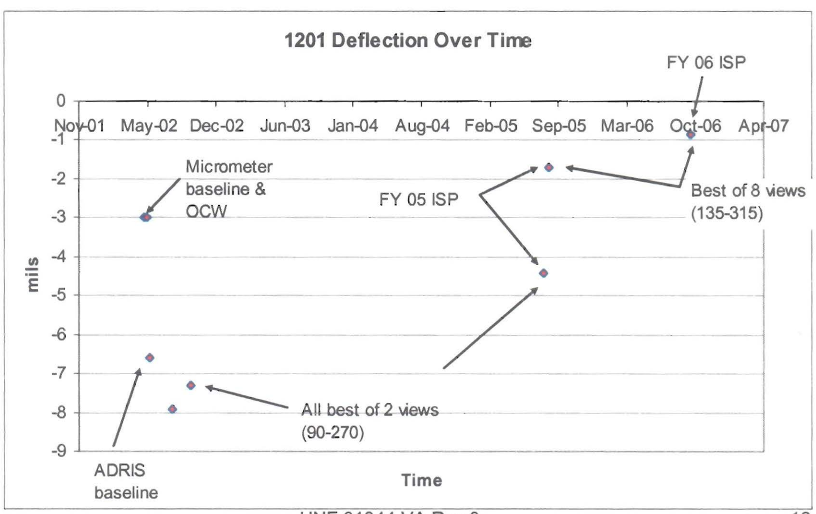
HNF-31944-VA Rev 0 10

Change in Deflection vs Reported Moisture



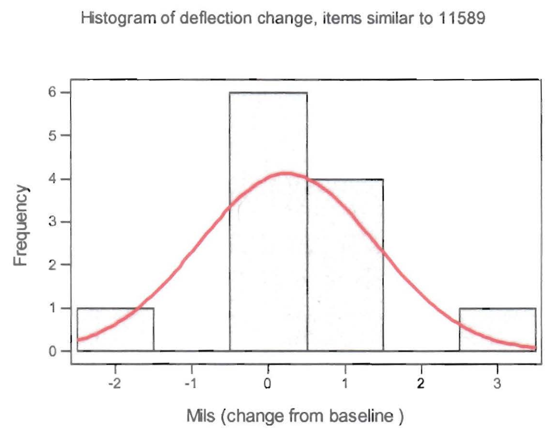
Container with highest overall change

- Fairly pure mixed oxide precipitated from mixed nitrate solution
- Water less than 0.25%, low net weight



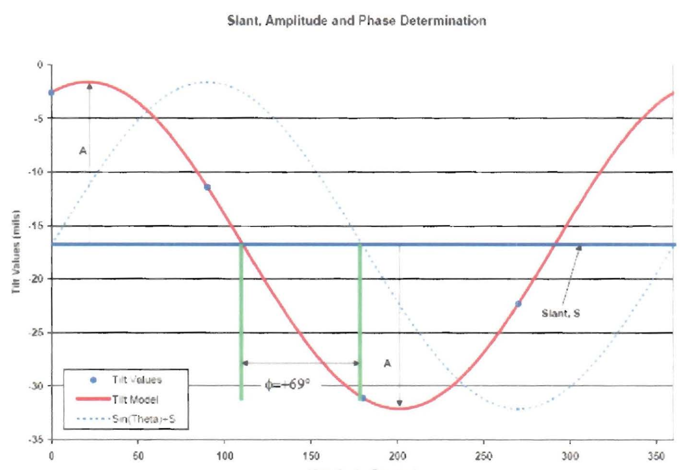
Items Similar to MIS item II589 Show no Detectable Pressure

- None was expected based on sample water % and can loading
- Each loading parameter about ½ of the Shelf-life test can



# of samples	13
Overall Average	+0.25
Deviation	1.2
95% Confidence Level	-0.5 to +1.0

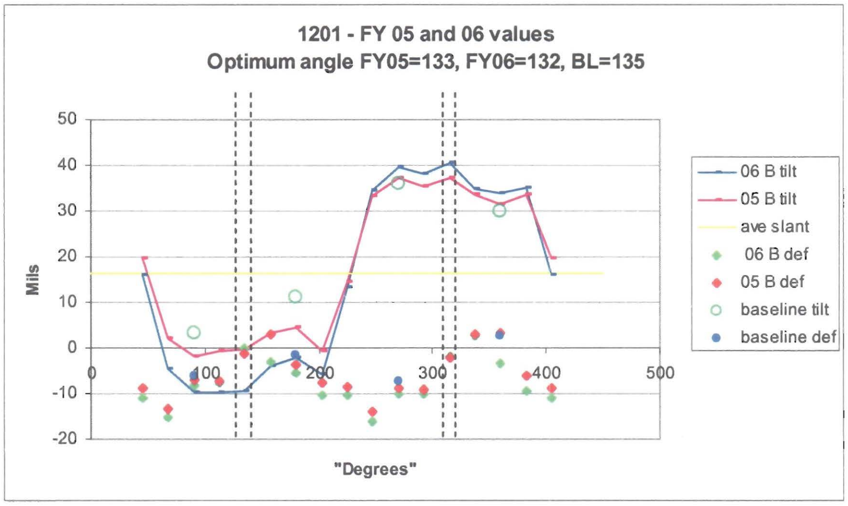
Radiography – Optimal View Model



Optimal View Model developed by SRS

Tilt values follow sinusoid pattern, with optimal angle found at maximum tilt

Tilt Values Follow Predicted Model



HNF-31944-VA Rev 0 15

Radiography Conclusions

- Generally no sign of detectable pressurization, similar to last years results
- Cans that exceed detection level limit are explainable
- Some resurvey recommended for next year
- None of the items in subgroup similar to MIS item 11589 show detectable pressurization, consistent with expectations
- ADRIS 8 angle radiography mitigates deadzone issue

HNF-31944-VA Rev 0 16

FY 07 Plans

- Continue with FY07 NDE, with selection of additional samples, repeat some FY06 samples
- Continue/Complete Prompt Gamma Analysis
  - Priority on items "potentially similar to 11589"
- Have requested DOE/RL guidance on ISP Destructive Analysis

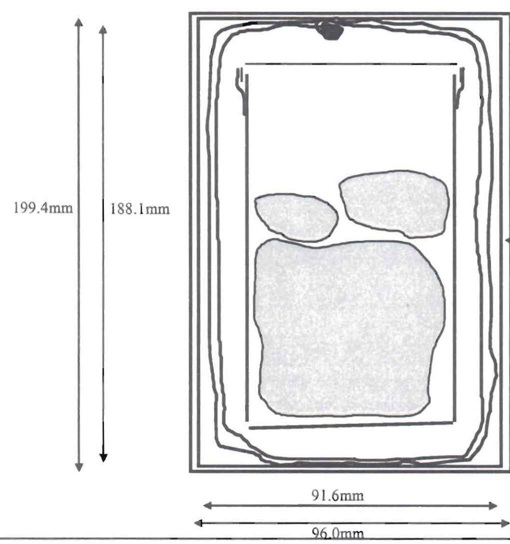
- Deinventory
  - Plan for 2 year delay, but also be ready to ship in Spring 2007
  - Establish 9975 annual maintenance leak test capability
  - Proceed with certification of un-irradiated fuel package (HUFP)

- Detailed FY06 Surveillance Report Issued – HNF-31775

AWE Storage Experiences

JOWOG 22
January 2007

ASC001 Package design



ASC001 Container
inner
outer
both rim sealed

DNFSB Paper

- "Sites continue to rely on container types that have been used historically, but have no technically justified safety or design basis"
- "The DOE should…determine the technically justified packaging criteria needed to ensure safe storage"

RAMSCAP

Radioactive and Special Materials Storage Containers Approval Panel

Approve the design requirements for packaging at AWE

Approve the use of package designs for storage

Main RAMSCAP Requirements

- Be designed to withstand credible internal pressures
- Accessible surfaces shall not exceed 85°C
- Have an assessment completed of package life taking into account nature of the payload
- Have inspection / surveillance programme as appropriate
- Be sealed and maintain leak rate of 5×10^{-5} $Pa.m^3s^{-1}$

AWE Design Test Requirements

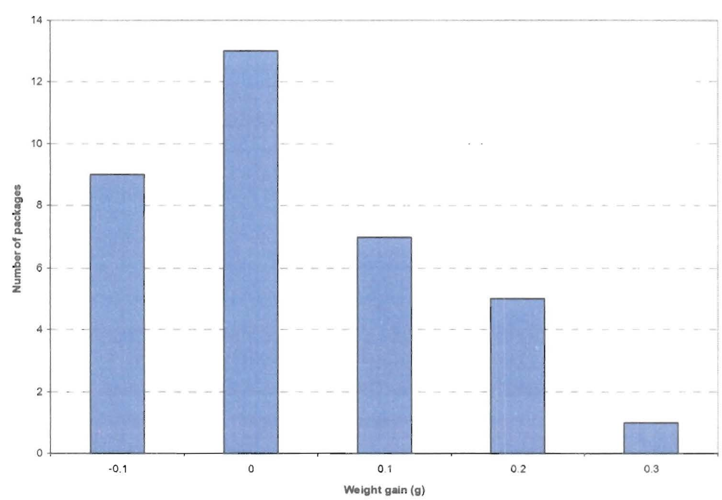
- Drop test from 1.2m onto base
- Drop test from 1.2m onto attitude likely to cause most damage
- Compressive load of 5 times the package mass
- Penetration test

- Required to maintain leak rate of 5×10^{-2} $Pa.m^3.s^{-1}$ after test

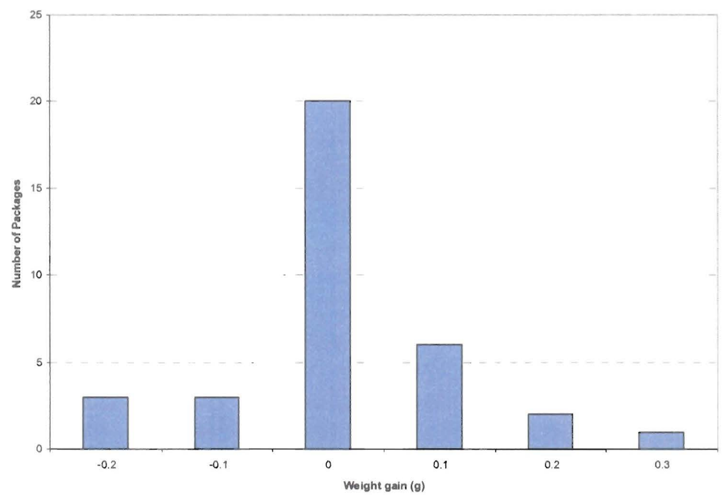
ASC001PO Package

- Approved for storage of plutonium oxide
  - produced from metal by an oxidation process at a minimum of 400°C in a gas stream containing at least 15% oxygen
  - cooled down and stored prior to packing in a dry atmosphere (<100vpm moisture)
  - material has passed through 800 micron sieve

Oxide weight changes



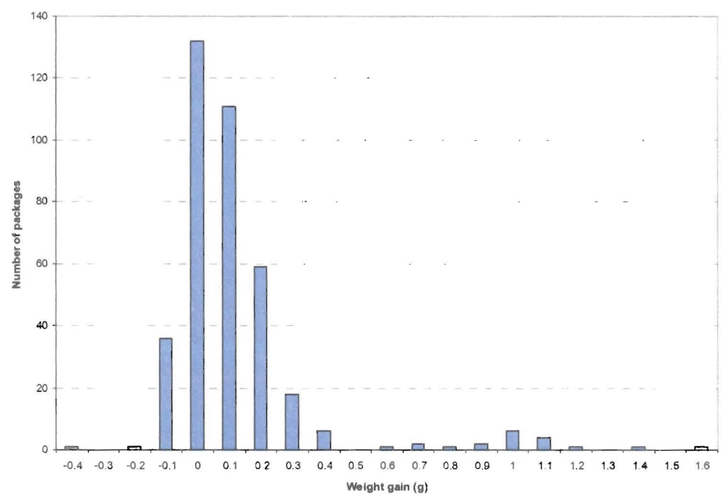
Oxide weight changes since last inspection



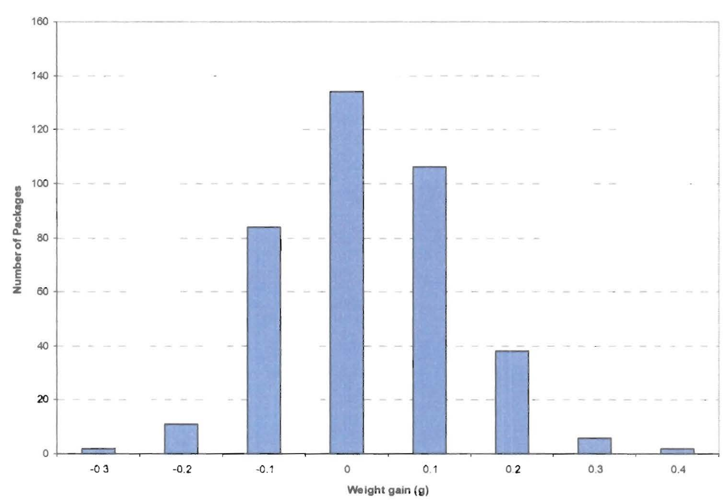
ASC001PI Package

- Approved for storage of salts
  - not metal or oxide
  - has either
    - been produced at greater than 400°C, or
    - been dried by an approved process at greater than 400°C
  - been cooled down and stored prior to packing in a dry atmosphere (<100vpm moisture)
  - does not contain significant organics

Salts weight changes



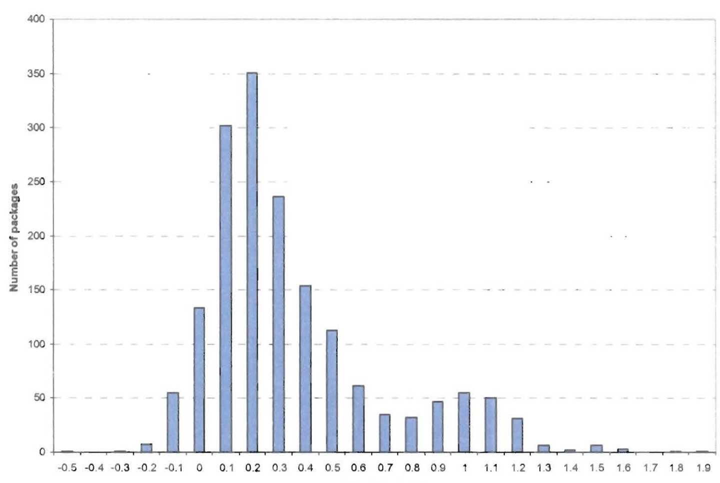
Salts weight changes since last inspection



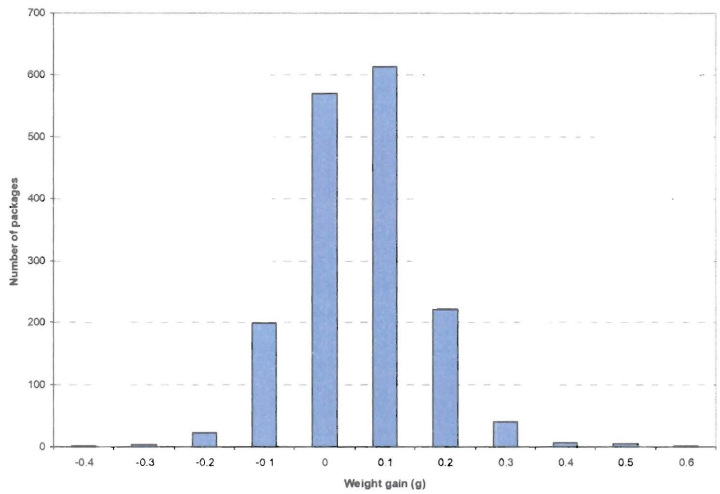
ASC001A Package

- Subgroup of ASC001PI package
- Contains materials which were not dried before being repacked
- Therefore required more frequent inspection programme

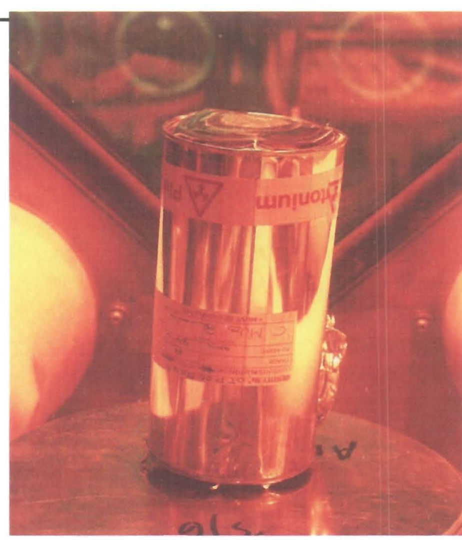
ER Salts weight changes



ER salt weight changes since last inspection



Pressurised can



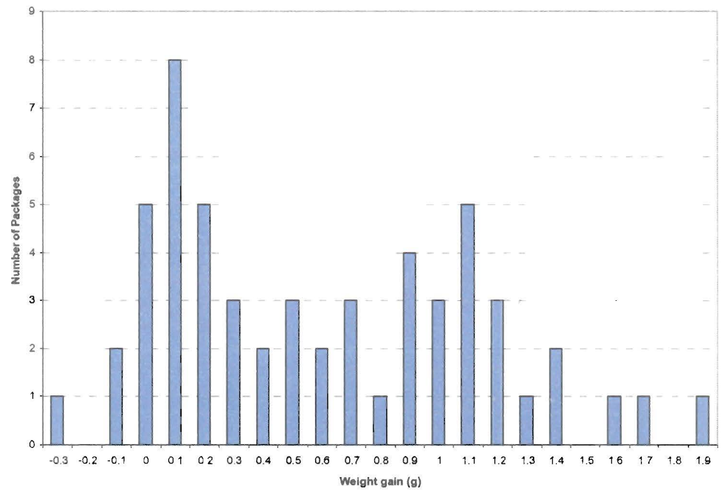
ASC001A package

- No pressurised packages seen for three years
- Inspection and surveillance did not indicate problems
- Approval now given to extend inspection frequency to three years as for other salts

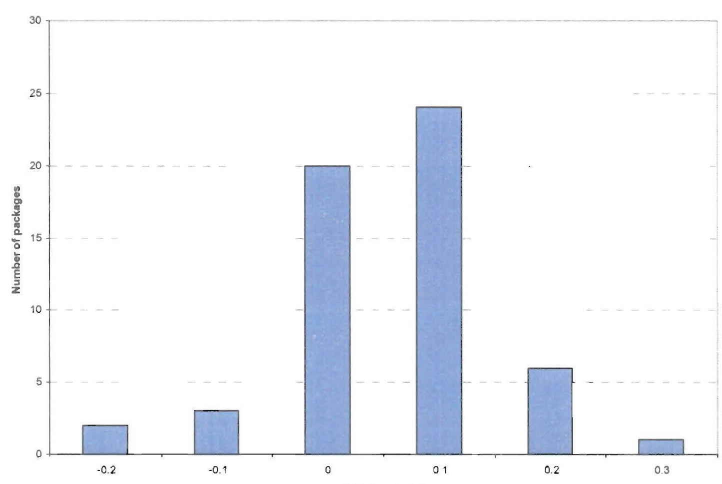
ASC001PM Package

- Approved for storage of metal
  - is metal in terms of provenance and appearance
  - is resilient
  - no loose powder adhering to the metal
  - no piece passes through a 1cm sieve

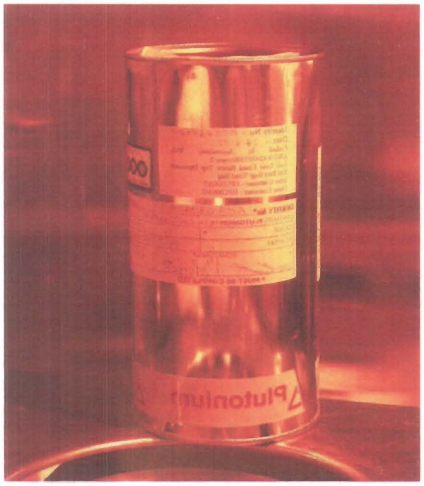
Metal weight changes



Metal weight changes since last inspection



Panelled Can



2001

2003

PVC Degradation

Discussion

- No packages failed resulting in loss of containment
- Weight gains seen are small
- Year on year weight gains are minimal, suggesting weight gains are not the result of moisture / oxygen absorption

Conclusions

- Storage packages are designed against published criteria
- There is an ongoing inspection and surveillance programme
- There have been no serious incidents since this regime was introduced

Rev 8 1-12-07
For technical review board review

MANUAL

DRAFT **DOE M 441.1-1**

Approved: XX-XX-XX
Expires: XX-XX-XX

NUCLEAR MATERIAL PACKAGING MANUAL

U.S. DEPARTMENT OF ENERGY
Office of Nuclear Safety and Environment

AVAILABLE ONLINE AT:
http://www.directives.doe.gov

INITIATED BY:
Office of Nuclear Safety and Environment

1. PURPOSE. This Manual provides detailed packaging requirements to protect workers from exposure to nuclear materials (stored outside of an approved engineered contamination barrier) whose composition and quantity pose the potential for a significant inhalation dose (i.e., 5 Rem committed effective dose equivalent). The requirements in this Manual are consistent with requirements in Title 10 Code of Federal Regulations Part 830 (10 CFR 830), *Nuclear Safety Management;* 10 CFR 835, *Occupational Radiation Protection*; and Department of Energy Policy (DOE P) 450.4, *Safety Management System Policy*; for evaluating hazards and identifying appropriate controls to protect facility workers.

   This Manual is not intended to conflict with or supersede accepted criteria established in 10 CFR 835 and in other applicable Department directives such as DOE Standard (DOE-STD)-3013, *Stabilization, Packaging, and Storage of Plutonium-Bearing Materials*; DOE-STD-3028, *Criteria For Packaging and Storing Uranium-233-Bearing Materials*; and DOE Handbook (DOE-HDBK)-1129, *Tritium Handling and Safe Storage.*

2. CANCELLATIONS. This supersedes Deputy Secretary of Energy Memorandum *Criteria for Interim Storage of Plutonium Bearing Materials* dated January 25, 1996.

3. APPLICABILITY.

4. SUMMARY. This Manual is organized into three chapters. Chapter I, *Overview and Responsibilities*, provides an overview of the manual and delineates responsibilities for packaging and storage of nuclear materials at the complex-wide and Field Element levels. Chapter II, *Scope of Material,* identifies the scope of materials for which the requirements of this manual are applicable. Chapter III contains detailed packaging requirements including surveillance and testing requirements.

5. REFERENCES. References are located in Attachment 3 of this Manual

6. CONTACT. Questions concerning this Manual should be addressed to Office of Health Safety and Security, Office of Nuclear Safety and Environment, at 202-586-6740.

SAMUEL W. BODMAN
Secretary of Energy

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CHAPTER I: OVERVIEW AND RESPONSIBILITIES

1. OVERVIEW.

Restates the Purpose in the Bodman Letter

2. RESPONSIBILITIES.

a. Program Secretarial Officers. Program Secretarial Officers with Nuclear Material Packaging facilities, operations, or activities are responsible within their respective programs for ensuring that their Field Element Managers meet the requirements in this Manual.

b. Office of Health, Safety and Security. The Chief Health Safety and Security Officer is responsible for:

(1) Safe Packaging for Nuclear Materials Storage

(a) Advising the Secretary of the status of Departmental compliance with the requirements in this Manual, and applicable provisions of related DOE Orders.

(b) Providing guidance and interpretation of this Nuclear Materials Packaging Manual.

(c) Reviewing and approving any requested exemptions to this Manual.

(2) **Changes to Regulations and DOE Directives.** Ensuring changes to regulations and DOE directives are reviewed and, when necessary, incorporated into revisions of this Manual to ensure the basis for safe storage of nuclear materials is maintained.

c. Field Element Managers. Field Element Managers are responsible for:

(1) **Review and approval of Nuclear Material Packaging Technical Basis.** Review and approve the contractor - developed technical basis for packaging systems

(2) **Surveillance Program.** Review of the contractor's nuclear materials packaging surveillance program and the implementation of the program.

(3) **Documentation.** Review of the contractor's process for documenting its nuclear materials storage program and the implementation of the process

(4) **Oversight.** Ensuring oversight of nuclear material packaging for storage facilities, operations, and activities is conducted in accordance with this Manual.

(5) **Corrective Actions.** Ensuring a process exists for proposing, reviewing, approving, and implementing site corrective actions.

(6) **Schedule for Implementation:** Establishing a risk-based prioritized schedule for implementing the nuclear material packaging requirements for material stored prior to the issuance of this Manual

d. Contractors. Contractors are responsible for implementing this Manual by developing a technical basis for their packaging system and implementing the packaging system after the Field Element Manager approves the technical basis document. The contractor is also responsible for implementing the CRD (Attachment 2).

CHAPTER II: SCOPE OF MATERIAL

1. Field Element Managers shall ensure that nuclear materials that are stored outside engineered containment barriers, and are of an isotopic composition and quantity such that, if released, could result in a 5 Rem or greater exposure to workers, are packaged in accordance with the detailed material packaging requirements described in Chapter III.

2. The following materials are not subject to the material packaging requirements of this Manual:

   a. Gases;

   b. Materials stored in approved packaging, such as described in DOE-STD-3013, DOE-STD-3028, and DOE-HDBK-1129;

   c. Materials designated as waste, which are transferred to appropriate waste containers in accordance with DOE O 435.1, *Radioactive Waste Management*, and all applicable facility specific requirements;

   d. Clad nuclear reactor fuels (fresh or spent);

   e. Nuclear material packaged for shipment in approved shipping containers in compliance with package specifications or Certificate of Compliance;

   f. Sealed sources as defined in 10 CFR 835.2;

   g. Material meeting the special form criteria in 49 CFR 173.403, *Definitions* and 49 CFR 469, *Tests for special form Class 7 (radioactive) materials*;

   h. Encapsulated (i.e. sealed or welded configurations) weapons components.

   i. Natural uranium (NU) and depleted uranium (DU); and

   j. Medical isotopes.

   k. Fixed contaminated or activated tools and equipment

In addition, the specific packaging criteria of this Manual are not applicable to material packaged for short durations in a bagged-out convenience container or other appropriate container while being transferred from one approved engineered contamination barrier to another or to the final package configuration.

CHAPTER III: NUCLEAR MATERIAL PACKAGING REQUIREMENTS

1. STORED MATERIAL CHARACTERISTICS CRITERIA.

Field Elements must ensure that the chemical, radiological, and physical characteristics of the material being stored are evaluated and are appropriate for the material package including the following:

a. Explosion Sensitive and/or Flammable Materials.

b. Gas Generation.

c. Incompatible Materials.

d. Physical and Chemical Form.

e. Moisture Content.

f. Pyrophoricity.

g. Radiation/Decay Heat.

h. Radiation Field.

i. Solutions.

2. PACKAGING DESIGN CRITERIA.

Field Elements must ensure that the nuclear material storage package meets the following design criteria.

a. General.

b. Package Container Types, Securing and Sealing.

(1) **Sealed Containers.**

(2) **Filter Vented Containers.**

(3) **Containers for Liquid Storage.**

(4) **Other Containers.** Plastic bags and slip-lid cans may be used inside the package but may not be counted as one of the storage containment barriers for high risk material (see Attachment 7 for the definition of high risk materials). Plastic containers shall not be in direct contact with alpha emitting high risk materials or alpha emitting low risk materials with a specific activity greater than one curie per gram (residual quantities of alpha emitting materials, e.g., residual contamination outside of inner

convenience packages, are allowed to be in contact with plastic). Use of plastic materials shall be appropriately analyzed according to Section III.2 and III.3. Processes and procedures that use plastic bags, bottles, or slip-lid cans must be documented prior to the usage of these materials as contamination barriers.

c. Corrosion Effects.

d. Filter Performance.

e. Heat Resistance.

f. Pressure Effects.

g. Pyrophoric Materials.

h. Radiation Resistance.

i. Design Leakage Rate.

j. Drop Test.

k. Design Qualification Leak Rate.

l. Liquids and Pyrophoric Materials.

m. Surveillance.

n. Fabrication.

o. Design Life.

p. Labeling.

q. Contamination Levels.

3. PACKAGING SURVEILLANCE PROGRAM.

Field Elements must ensure that a surveillance program is established and implemented to ensure the nuclear material storage package continues to meet its design criteria including the following elements.

a. General.

b. As Low As Reasonably Achievable (ALARA).

c. Objectives and Techniques.

(1) Visual inspection

(2) Weight measurement.

(3) Contamination surveys

(4) Testing of vent filters

(2) Radiography

(3) Opening container for examination of interior and contents

d. Frequency.

e. Procedures.

f. Evaluation.

4. PACKAGING DOCUMENTATION.

Field Elements must ensure that documentation of the nuclear material storage package design and surveillance is maintained.

a. Records Retention.

b. Content Elements.

(1) Material Information.

(a) Chemical and physical form;

(b) Best available isotopic content, and the effective dates(s) of analysis;

(c) Weight of material contents; and

(d) Description of container contents.

(2) **Package Information.**

(a) Package configuration;

(b) Date of packaging for each container;

(c) Baseline package gross weight; and

(d) The unique identification number associated with each container.

(3) **Surveillance Information.**

(a) The unique identification number;

(b) Surveillance and radioactive survey results and dates;

(c) Dates, location, and results of inspections; and

(d) Name and site qualifications of individuals performing inspections.

(4) **Technical Basis Information.**

c. Database.

ATTACHMENT I: ALL DEPARTMENT ELEMENTS TO WHICH DOE M 441.1 IS APPLICABLE

ATTACHMENT II: CONTRACTOR REQUIREMENTS DOCUMENT

ATTACHMENT III: REFERENCES

ATTACHMENT 4: METHOD FOR DETERMINING TYPES AND QUANTITY OF MATERIAL SUBJECT TO SPECIFIC PACKAGING DESIGN REQUIREMENTS

This attachment provides an acceptable method for determining the types and quantities of stored material that could result in an internal exposure to workers above 5 Rem (CEDE) if not properly packaged and therefore are subject to the specific packaging design requirements of this Manual.

1. TYPES OF MATERIAL.

   Table 1, *Applicable Isotopes*,

2. QUANTITIES OF MATERIAL.

   Nuclear materials that are of the type described above and where the total activity exceed the A_2 thresholds 49 CFR 173.435, *Table of A_1 and A_2 values for radionuclides*, are subject to the specific container requirements of this Manual. The thresholds are utilized in 49 CFR 173.435 to identify requirements for packaging of material for transport of public roadways. For mixtures of isotopes, sites shall use a methodology consistent with that in 49 CFR 173.433.

3. METHODOLOGY FOR PERFORMING THRESHOLD CALCULATIONS.

   Threshold calculations can be used to determine whether a material is in or out of scope, and if in scope, if the material is low or high risk. Thresholds are defined as one times A_2 ($1xA_2$) for low risk and twenty times A_2 ($20xA_2$) for high risk. Threshold Calculations shall use one of the following methodologies.

   a. The quantity of material to be packaged shall be compared to Table 1. The A_2 value can be used directly from Table 1 using either the A_2 curie threshold value or the 5 rem CEDE or the 100 rem CEDE threshold values in grams. When obtaining Table 1 quantities, nuclides that are parents of decay chains with daughters with > 10 day half lives must be considered as mixtures with the longer lived daughters, and the mixture equation in 49 CFR 173.433(d) must be applied. For example, uranium enriched greater than 20% must be treated as a mixture of parent nuclides with all daughters with half lives greater than 10 days. If the identities of the radionuclides in a mixture are in doubt, the lowest A_2 values with the total quantity of material shall be used for the mixture.

   b. Threshold calculations of any dispersable material containing a single radionuclide or a mixture of radonuclides shall be determined using 49 CFR 173.433, *Requirements for determining basic radionuclide values, and for the listing of radionuclides on shipping papers and labels.*

Materials in "solid form" do not have to be included in the threshold calculation. Material considered to be solid form must meet the following:

a. Solid forms are greater than 100 um AED at initial packaging.

b. Solid forms maintain AED greater than 100um after a post drop event.

c. Quantities of material less than 100 um formed during storage due to corrosion, etc. or upon material disruption (container drop) must be included in the threshold calculation.

In determining whether material is solid form calculations must consider reactions that could generate powders (i.e. oxidation, decay heat, and physical degradation) over the life time of the storage conditions. For example, a container that has a large metallic object with very little (less than an A2 quantity) of powder may not be required to meet the specific container requirements of this Manual if it can be further demonstrated that less than an A2 quantity of fine material will result during the Design Life of the container due to reactions during storage or during a drop event.

Table 1 DOE M 441.1 Threshold Quantities for Applicable Isotopes**

Isotopes	A_2 (Ci)	Specific Activity (Ci/g)	Low Risk 5 rem CEDE Quantity (g)	High Risk 100 rem CEDE Quantity (g)
U-232	$2.7x10^{-2}$	$2.2x10^{1}$	$1.2x10^{-3}$	$2.4x10^{-2}$
U-233	$1.6x10^{-1}$	$9.7x10^{-3}$	$1.6x10^{1}$	$3.2x10^{2}$
U-234	$1.6x10^{-1}$	$6.2x10^{-3}$	$2.6x10^{1}$	$5.2x10^{2}$
U-235* [a]	Unlimited	$2.2x10^{-6}$	Unlimited	Unlimited
U-235 enriched < 20%	Unlimited	$2.2x10^{-6}$	Unlimited	Unlimited
U-235 enriched > 20%	**	$2.2x10^{-6}$	**	**
U-236	$1.6x10^{-1}$	$6.5x10^{-5}$	$2.5x10^{3}$	$4.9x10^{4}$
U-238*	Unlimited	$3.4x10^{-7}$	Unlimited	Unlimited
Pu-238	$2.7x10^{-2}$	$1.7x10^{1}$	$1.6x10^{-3}$	$3.2x10^{-2}$
Pu-239	$2.7x10^{-2}$	$6.2x10^{-2}$	$4.4x10^{-1}$	8.7
Pu-240	$2.7x10^{-2}$	$2.3x10^{-1}$	$1.2x10^{-1}$	2.4
Pu-241	1.6	$1x10^{2}$	$1.6x10^{-2}$	$3.2x10^{-1}$
Pu-242	$2.7x10^{-2}$	$3.9x10^{-3}$	6.9	$1.4x10^{2}$
Th-228	$2.7x10^{-2}$	$8.2x10^{2}$	$3.3x10^{-5}$	$6.6x10^{-4}$
Th-229	$1.4x10^{-2}$	$2.1x10^{-1}$	$6.7x10^{-2}$	1.3
Th-232*	Unlimited	$1.1x10^{-7}$	Unlimited	Unlimited
Np-237	$5.4x10^{-2}$	$7.1x10^{-4}$	$7.6x10^{1}$	$1.5x10^{3}$
Am-241	$2.7x10^{-2}$	3.4	$7.9x10^{-3}$	$1.6x10^{-1}$
Am-243	$2.7x10^{-2}$	$2x10^{-1}$	$1.4x10^{-1}$	2.7
Bk-249	8.1	$1.6x10^{3}$	$5.1x10^{-3}$	$1.0x10^{-1}$
Cm-244	$5.4x10^{-2}$	$8.1x10^{1}$	$6.7x10^{-4}$	$1.3x10^{-2}$
Cm-246	$2.7x10^{-2}$	$3.1x10^{-1}$	$7.7x10^{-2}$	1.6
Cf-252	$8.1x10^{-2}$	$5.4x10^{2}$	$1.5x10^{-4}$	$3.0x10^{-3}$

*The quantities for U-235, U-238, and Th-232 require an intake of more than 10 mg to result in the given threshold dose. Standards established by the IAEA assume an individual will inhale no more than 10 mg.

For uranium isotopes, nonradiological effects, such as chemical toxicity, may be limiting and require evaluation per 10 CFR 851 and DOE O 440.1A.

**Gram values are based on 49 CFR 173.435 dated October, 2005. Isotopic mixtures vary and shall be calculated. Refer to 49 CFR 173.433.

[a] These isotopes have A_2 values that considered the contributions from progeny with less than 10 day half-life.

Notes:

1. Table 1 also provides thresholds of material quantities which meet the definitions of High Risk and Low Risk materials. High Risk materials are defined as those materials with quantities of radionuclides that if released could result in exceeding 100 Rem CEDE, and Low Risk materials that if released could result in exposure between 5 rem CEDE and 100 rem CEDE. Thresholds are defined as one times A_2 ($1xA_2$) for low risk and twenty times A_2 ($20xA_2$) for high risk
2. Materials below 5 rem CEDE are below the A_2 values defined in 49 CFR 173.435 and are out of scope for the specific packaging design requirements of this Manual.
3. The methodology and assumptions used in calculating the A_2 quantities in 49 CFR 173.435 are also the same for the high and low risk thresholds. The A2 quantities in 49 CFR 173.435 were determined from a calculation of the amount of packaged material which result in a 5 rem dose if 10^{-3} of the material became airborne as a result of the storage, handling or accident and 10^{-3} of the airborne material was subsequently inhaled and incorporated into a person located in the vicinity of the material. This is based upon analysis by IAEA.
4. 100 Rem CEDE was chosen as the threshold for high risk materials because (1) it is below the 200 Rem CEDE where significant health effects occur, (2) it is consistent with values used at some DOE sites for determining whether systems protecting facility workers should be designated as safety significant to meet 10 CFR 830 requirements.
5. Table 1 is not all inclusive of all isotopes that meet the criteria of inhalation dose exceeding the external dose. Sites must evaluate the storage of uncommon isotopes to ensure that the specific packaging requirements of this Manual (i.e., inhalation dose exceeding and potential for greater and exceeding quantity of A_2 values in 49 CFR 173.425) are met.

ATTACHMENT 5: METHODOLOGY AND CRITERIA FOR MEETING LEAK RATE REQUIREMENTS

This attachment describes a methodology and acceptance criteria that are acceptable for meeting this Manual's requirements for (1) design leak rate and (2) design qualification leak rate.

1. DESIGN LEAK RATE.

The design leak rate is intended to correspond to potential leakage of undisturbed material in storage packages and accounts for changes in atmospheric pressure and minor buildup of pressure corresponding to 1 atmosphere differential pressure. This design leak rate is consistent with leak rate criteria for Type B packages as described in ANSI N 14.5-1997.

a. Acceptance Criteria. All of the following criteria result in high assurance that potential exposure to a worker is maintained ALARA and will not receive a dose exceeding his/her annual exposure limits.

(1) Gas Leak rate from the container less than $1x10^{-3}$ cm^3/s (Test of gas subject to 1 atmosphere differential pressure; [see Note 1]

(2) Particulate release of 10^{-3} A_2/hour; [see Note 2]

(3) Facility specific particulate leak rate determined such that releases are maintained ALARA and exposures to workers below 5 rem CEDE (Determined via analysis or testing of surrogate material). [See Note 3]

b. Testing methodology. The ANSI 14.5-1997 test methodology should be used for the gas leak rate test.

For the particulate release rate test, the test shall simulate normal handling condition (include movement of the package with accompanying disturbance of contained material during movement)

2. DESIGN QUALIFICATION LEAK RATE.

a. Acceptance Criteria. All of the following criteria result in high assurance that potential exposure to a worker is maintained ALARA and will not receive a dose exceeding his/her annual exposure limits following a drop of a nuclear material package.

(1) Gas Leak rate less than $1x10^{-3}$ cm^3/s ;

(2) Particulate Release of 10^{-3} A_2/event; [See Note 4]

(3) Facility specific particulate leak rate determined such that releases are maintained ALARA and exposures to workers below 5 rem CEDE (Determined via analysis or testing of surrogate material). [See Note 5]

b. Testing methodology. The ANSI 14.5-1997 test methodology should be used.

Notes:

1. The basis for the 10^{-3} cm^3/sec value for the design leak rate is that it is consistent with ANSI N14.5's acceptable pre-shipment leak rate of 10^{-3} ref-cm^3/sec (Section 7.6.4). Furthermore, 49 CFR 178, *Specifications for Packagings*, has leak testing requirements. Subpart M, *Testing of Non-bulk Packagings and Packages*, has a leak tightness (49 CFR 178.604) and a hydrostatic pressure test (49 CFR 178.605). Although these are not for radioactive material containers, they do provide reasonable criteria for leak tightness.

   The leak tightness test in the referenced documents is to pressurize the container with 4 psig, submerge the container in water and see if any bubbles are formed as the gas leaks from the container. ANSI 14.5 Appendix A states that a bubble test has a nominal test sensitivity of 10^{-3} ref cm^3/sec.

2. The basis for the design qualification leak rate less than 10^{-3} A_2/hr is that a release of this amount during handling for an hour would not result in exceeding 5 rem to the worker (the A_2 values were derived based an assumption that 10^{-3} of the material is released).

3. The basis of this criterion is that it meets the fundamental objective of the Manual while providing flexibility for users to define specific criteria based upon facility specific conditions. The site-specific criteria should be justified as to the degree of conservatisms.

4. The basis for the design qualification leak rate less than 10^{-3} A_2/event is that a release of this amount during an event would not result in a dose exceeding 5 rem to the worker (the A_2 values were derived based an assumption that 10^{-3} of the material is released)

5. The basis for the design qualification leak rate less A_2/wk is that this is consistent with the ANSI 14.5 criterion, it is equivalent to $1.65x10^{-6}$ A_2 per second, and not will result exposure to the worker above 5 Rem.

3. DROP TEST.

The drop test should be performed in accordance with ANSI 14.5-1997.

Note:

For the purpose of this Manual additional conservatism was provided above that specified in ANSI 14.5-1997 in that the testing height is 1.5 times the maximum working height (for high risk material) and 1.2 times the maximum working height (for low risk material). This conservatism was deemed appropriate to address potential variations in packaging capabilities and testing uncertainties. The higher value for the higher risk material is consistent with DOE graded approach.

ATTACHMENT 6: ACRONYMS

ATTACHMENT 7: DEFINITIONS